

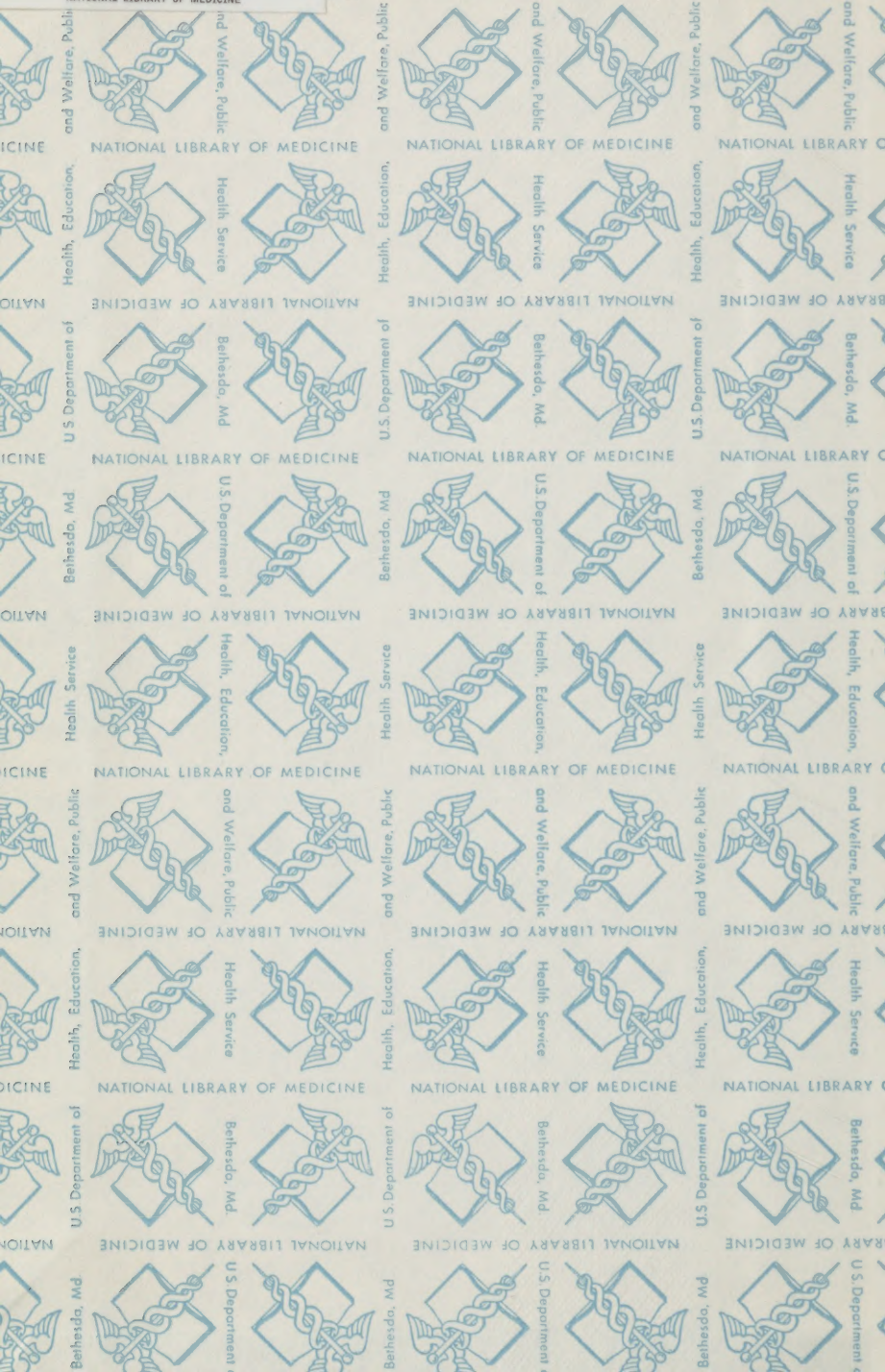
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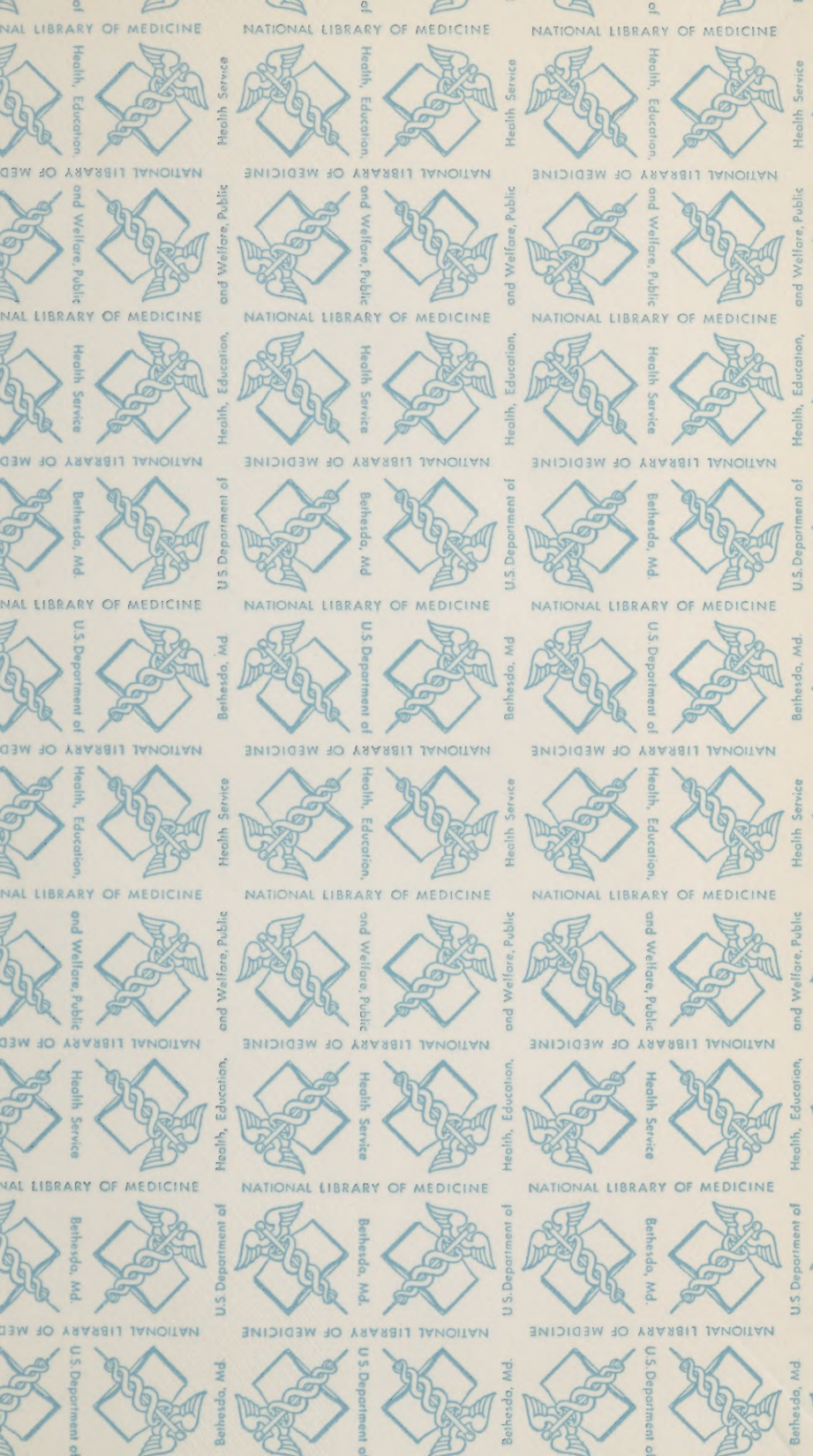
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PRACTICAL AND ANALYTICAL
CHEMISTRY.

HENRY TRIMBLE, PH.G.



PRACTICAL AND ANALYTICAL CHEMISTRY.

BEING

A COMPLETE COURSE IN CHEMICAL
ANALYSIS.

✓ BY

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ILLUSTRATED.



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P R E F A C E.

The increased amount of time devoted by students of pharmacy and medicine to analytical chemistry has directed more attention to the subject of imparting instruction in this science.

The object of the present volume is to place before the student as compact a course as possible, in order to enable him to become familiar with the subject in the necessarily limited time at his disposal.

The author's experience has led him to believe that a study of Qualitative Analysis should be preceded by some experience in the preparation of the more important gases and a few of the salts. Such practice requires the student to familiarize himself with the construction of apparatus as well as with the processes of filtration, evaporation, crystallization, ignition, etc.

The examples for preparation may be increased at the option of the instructor.

In Part II the student should perform the reactions of each group, and then be furnished with a solution containing some, or all, of the bases of the group. This should be followed by a solution in which he should search for all the elements previously studied; such practice being repeated until he can correctly determine all the bases present.

In order to enable the student to see the comparative effect of the group reagents readily, a summary has been introduced at the end of each group. This he should be able to write

out without the use of the book before attempting to analyze a group solution. By progressive steps he is thus led rapidly on to successfully examine the more complex solutions for both bases and acids.

The grouping of the bases is to a certain extent new, but it places together those elements which are very closely related, and, in addition, adapts each group to the time of one lesson, which may be repeated if desirable.

In the Part devoted to gravimetric and volumetric analysis, the examples are limited in number; but this much is intended to give the student an opportunity to learn the methods with the aid of an instructor, so that he may afterwards pursue the subject alone, with the aid of a book like Fresenius's Quantitative Analysis.

In conclusion, it is but just to state that many of the works on Qualitative Analysis have been consulted.

Those of Attfield and Muter furnished many valuable hints, but Fresenius's admirable work has been used as authority, and, to those who wish to pursue the subject in greater detail than here offered, it is recommended for reference.

H. T.

Philadelphia, August 19th, 1885.

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PART FIRST.

PRACTICAL.

PRACTICAL AND ANALYTICAL CHEMISTRY.

PART I.—PRACTICAL.

SECTION I.

PREPARATION AND PROPERTIES OF GASES.

HYDROGEN, H.

Preparation.—Place a few fragments of zinc in a flat-bottom flask of about one-fourth liter capacity; cover the zinc with water and adapt a cork, through which pass two tubes (Fig 1), one just reaching through the cork and bent so the long end may be dipped under water, the other running directly from a short distance above the cork nearly to the bottom of the flask, so as to be below the surface of the liquid. The upper end of this tube should have a small funnel placed in it, or be enlarged by softening in the flame, inserting and revolving a file or similar instrument, previously warmed.

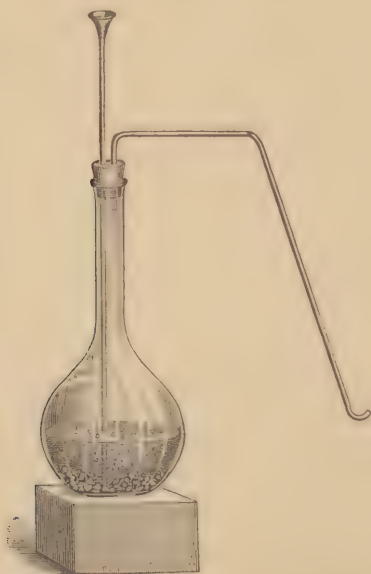
Add, slowly, a small quantity of sulphuric acid, through this tube, and notice an immediate effervescence, with the escape of bubbles through the water in which the exit tube dips. Fill a test tube with water, and, keeping the open end under the liquid, bring it over the tube, so as to collect the gas. When full, close with the thumb, and, bringing the mouth of the tube near a flame, quickly remove the thumb and allow the gas to ignite. It will burn quietly if the gas be pure, but with a slight explosion if it be mixed with air.

Properties.—This gas is **Hydrogen**, and its physical properties may now be studied by observing that it is insoluble in water, and without odor, color or taste.

EXPERIMENT I. Collect a tube full, and holding it, covered, in a vertical position, bring a lighted taper a short distance above its mouth and remove the cover; the gas will ignite, showing its great levity.

EXPERIMENT II. Another tube, similarly filled, is held in an inverted position, and the cover removed; it will be found, even after the lapse of some time, that the hydrogen at the mouth of the tube may be ignited, thus demonstrating that the gas is too light to come down and out the mouth of the tube. These two experiments have also demonstrated the combus-

FIG. 1.



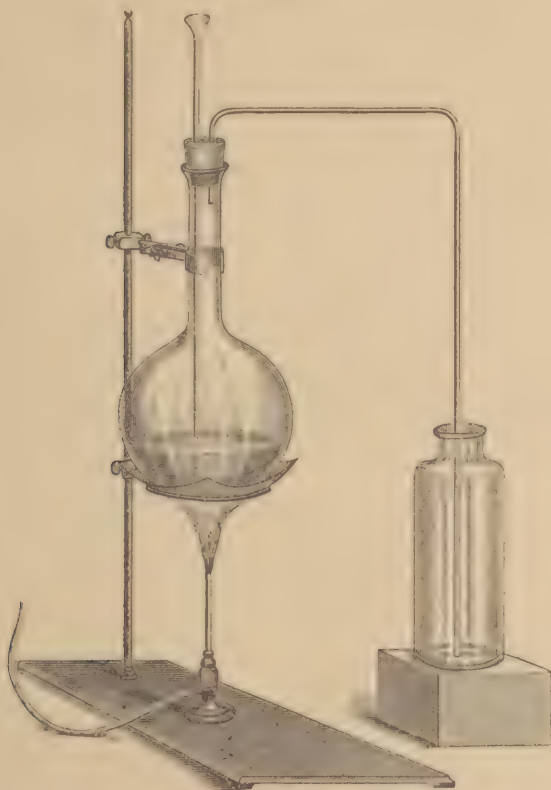
tibility of the gas, which, when pure, burns quietly, with a colorless flame. If, however, it be mixed with air and flame applied, a violent explosion ensues. *Therefore, the tube from the generator should never be brought near a flame until it is certain all the air has been expelled.* This is determined by trying a test tube full; if it burn quietly, the jet may be lighted. *This precaution should always be observed.*

EXPERIMENT III. On bringing a lighted taper to the mouth of a tube full of hydrogen, the gas is ignited, but on pushing the burning taper up into the gas its flame is extinguished,

thus showing that while hydrogen is combustible it is not a supporter of combustion.

EXPERIMENT IV. Continue the addition of acid to the zinc until the latter is nearly all dissolved; disconnect the apparatus, pour the liquid on a filter, collect the filtrate in a small beaker or evaporating dish, concentrate and set aside for twenty-four hours, to crystallize. These crystals are *sinc*

FIG. 2.



sulphate, ZnSO_4 , the result of a combination of the sulphuric acid and the zinc, as follows:—



CHLORINE, Cl.

Preparation.—In a flask, arranged so that heat may be applied (Fig. 2), place a small quantity of manganese dioxide,

add hydrochloric acid, agitate well, to moisten all the powder on the bottom, and apply heat. A yellowish-green gas is evolved, which being somewhat soluble in water may be collected by downward displacement, that is, by running the delivery tube to the bottom of the receptacle, loosely covered, the heavy gas displaces the lighter air.

If the evolution be moderately active it may be collected over warm water, as only a small loss occurs. Care should be taken to avoid inhaling the gas, by passing it, when not collecting, into a solution of potassium or sodium hydrate. The following expresses the reaction in symbols:—

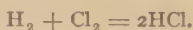


Properties.—EXPERIMENT I. Pass the gas into water, it is absorbed; if this be continued until the water is saturated, it will be found to have absorbed about twice its volume of the gas; the *Aqua Chlori* of the Pharmacopœia is the resulting product.

EXPERIMENT II. A tube full of the gas held with mouth upward, and a lighted taper applied, fails to ignite. Push the taper into the gas; it is extinguished, or only burns with a small, dense, smoky, flame, the result of a combination of the chlorine with the hydrogen of the wax, liberating the carbon.

HYDROCHLORIC ACID, HCl.

Preparation.—Collect one test tube full of hydrogen and one of chlorine, bring their mouths together (the hydrogen tube above with mouth down, as it is lighter), turn over once or twice, so as to thoroughly mix, and open their mouths to a flame; a sharp report will occur, with the development of strongly acid fumes, which will be recognized by future tests as *hydrochloric acid*:—



To prepare a quantity of it the apparatus used for the preparation of chlorine serves best. Put into the flask some sodium chloride (common salt), add sulphuric acid slowly and when the evolution of gas ceases apply a gentle heat. Collect by downward displacement or over mercury:—



The above expresses the reaction when an excess of acid has been used, which is preferable, as the resulting acid sodium sulphate is easily dissolved out of the flask with water. On the large scale the following more economical method is used:—



Properties.—The pungent, suffocating odor and freedom from color are noted with its production.

EXPERIMENT I. A test tube or jar of the gas placed with the open mouth under water will so rapidly dissolve that the liquid rises in the vessel. This solution of the gas in water, when of the proper strength, is the *Acidum Hydrochloricum*, U. S. P.

EXPERIMENT II. A piece of moistened blue litmus paper held in a tube of the gas is instantly reddened.

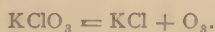
EXPERIMENT III. A lighted taper applied to the gas fails to ignite it, and is extinguished if lowered into it.

EXPERIMENT IV. Bring a rod moistened with ammonia over the mouth of a tube full of the gas; dense white fumes of ammonium chloride are formed.

The mixture remaining in the flask dissolved in warm water, treated with sodium carbonate so long as effervescence occurs, to neutralize the excess of sulphuric acid, concentrated, filtered and set aside to crystallize, yields *sodium sulphate* (Glauber salt).

OXYGEN, O.

Preparation.—Place a few crystals of potassium chlorate in a test tube, adapt a delivery tube long enough to reach under the surface of some water near by. Apply a steady flame; as soon as the bubbles of gas escape freely and the air has been expelled, bring a test tube filled with water over the escaping gas and collect. It is **Oxygen**, produced from the potassium chlorate by heat, according to the following reaction:—



Properties.—The appearance and insolubility of the gas in water are noted as it is collected.

EXPERIMENT I. The gas is not ignited by the application of a lighted taper. The taper, however, will burn with greatly

increased energy if it be plunged into the gas. If the flame be extinguished and the taper again brought into the gas, provided a spark remain, the taper is rekindled.

EXPERIMENT II. A piece of charcoal previously ignited is lowered into the gas; a rapid combustion ensues, and the charcoal disappears. Pour some lime water into the tube, agitate well; a white precipitate of calcium carbonate is produced. If this be tried with oxygen previous to the burning of the charcoal, no precipitate will be formed. A number of other substances, as sulphur, phosphorus and even iron when once kindled, will burn in oxygen with great brilliancy, forming characteristic oxides.

NITROGEN, N.

Preparation.—The usual method of preparing this gas is to deprive air of its oxygen, leaving the nitrogen pure. This is best accomplished by placing a small fragment of phosphorus on a cork covered with some fireproof material. Float the cork and phosphorus on water, ignite the latter and bring over it a bell-jar. The phosphorus combines with the oxygen, converting it into phosphorus pentoxide, P_2O_5 , which dissolves in the water present, thus leaving the nitrogen pure. A better method for obtaining larger quantities is to heat a mixture of potassium nitrite and ammonium chloride dissolved in water. When the reaction begins the temperature must be carefully watched, in order to prevent the too rapid evolution of the gas:—



Properties.—The physical properties have been observed during its preparation and collection. In regard to chemical properties, it is inert in the free state. Its compounds, however, are very energetic.

AMMONIA, NH_3 .

Preparation.—In a test tube or evaporating dish mix equal quantities of powdered calcium oxide (quicklime) and ammonium chloride; the odor of ammonia will be immediately developed:—



In smaller quantities the gas may be recognized by holding over the mixture a strip of moistened red litmus paper, it will slowly become blue, or similarly hold a glass rod moistened with hydrochloric acid, dense white fumes of ammonium chloride will form. To prepare larger quantities of the gas, heat the ordinary water of ammonia, which at a comparatively low temperature gives it off freely. If it be desired to dry the gas it must be passed over quicklime. Ammonia is collected by *upward displacement*, that is, by passing the delivery tube upward into a jar or test tube inverted over it; being lighter than air the latter is displaced.

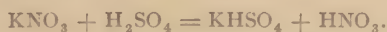
Properties.—EXPERIMENT I. Place a vessel filled with ammonia gas, mouth downward, into some water, and agitate slightly; the water will rise in the vessel rapidly, nearly filling it, showing the great solubility of the gas in water. The other physical properties, as color, odor, etc., have been noted during its collection.

EXPERIMENT II. On applying a lighted taper to the gas it does not burn; if, however, it be mixed with oxygen it will ignite readily. On introducing the taper into the gas it is extinguished.

NITRIC ACID, HNO_3 .

Preparation.—Place a small quantity of potassium nitrate in a test tube, and cover it with strong sulphuric acid. Apply a gentle heat; brown, strongly acid fumes are given off. To prepare a larger quantity a retort is used, to which is adapted a glass receiver. The nitric acid distils over on the application of a moderate heat, forming a reddish-yellow liquid, which rapidly attacks and destroys organic matter.

Two reactions may be employed to represent the production of nitric acid, depending on the relative quantity of the materials used. In the first case an excess of sulphuric acid gives—



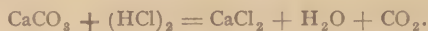
In the second case just a sufficient amount of sulphuric acid is used to decompose the potassium nitrate—



On the large scale sodium nitrate is now employed in place of the potassium salt, on account of its lower price.

CARBON DIOXIDE, CO_2 .

Preparation.—The flask used in the preparation of hydrogen will serve for making carbon dioxide. A few pieces of marble are placed in the flask, covered with water, and hydrochloric acid added. A brisk effervescence ensues, and the gas being somewhat soluble in water is collected by downward displacement—



Sulphuric acid should not be used, as it forms an insoluble calcium sulphate which is very difficult to remove from the flask.

Properties.—**EXPERIMENT I.** Pour some clear lime water into a jar of the gas and agitate; the solution immediately becomes cloudy, owing to formation of insoluble calcium carbonate. If more gas be passed into the mixture it will become clear again, on account of the solubility of the precipitate in carbonic acid.

EXPERIMENT II. Add solution of potassium hydrate to a jar of the gas, close and shake well; the gas is absorbed by the alkali, as may be shown by placing the mouth of the jar under water and removing the stopper, when the water will rush in, nearly filling it.

EXPERIMENT III. A lighted taper lowered into the gas is immediately extinguished. The same result is accomplished by opening the vessel some distance above the flame and allowing the gas to flow down upon it. This latter experiment also illustrates the great density of the gas, which is twenty-two times heavier than hydrogen.

SECTION II.

PREPARATION OF SALTS.

POTASSIUM CHLORIDE, KCl.

Preparation.—One or two cubic centimeters of hydrochloric acid, diluted with three or four times its bulk of water, are placed in a small beaker glass, and potassium carbonate added so long as effervescence occurs, and until after boiling the solution is neutral to litmus paper, that is, when the blue litmus paper is not changed to red nor the red changed to blue. Evaporate to a small bulk and set aside to crystallize. The cubic crystals which separate after standing twenty-four hours may be collected on filter paper and dried at a moderate temperature:—



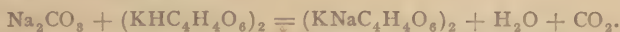
Potassium chloride is rarely prepared in this manner, except for practice, as it occurs largely in nature, and is used for preparing many other potassium salts.

POTASSIUM AND SODIUM TARTRATE,



(ROCHELLE SALT.)

Preparation.—Heat, in a porcelain capsule, a solution of sodium carbonate, and add to it potassium bitartrate until effervescence ceases, and the solution (after the escape of CO_2) is neutral to litmus. On filtering and cooling, crystals of Rochelle salt are deposited, rapidly and in small crystals if the solution be concentrated, but slowly and in much larger ones if the solution be dilute:—



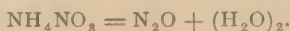
Note on Calculation.—In order to calculate the amount of each salt to use in the above process, we notice the number of molecules of each employed, and multiply this by the sum of the atomic weights (that is, by the molecular weights). In the above case one molecule of $\text{Na}_2\text{CO}_3 = 106$, and two molecules of $\text{KHC}_4\text{H}_4\text{O}_6 = 2 \times 188.1 = 376.2$. Therefore every 106 parts of anhydrous sodium carbonate require 376.2 parts of potassium bitartrate, to form Rochelle salt. If we have 50 grams of anhydrous sodium carbonate and wish to convert it into Rochelle salt, we use the following formula: As $106 : 376.2 :: 50 \text{ grams} :$ number of grams of potassium bitartrate required $= 177.4 \text{ grams}$.

AMMONIUM NITRATE, NH_4NO_3 .

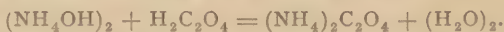
Preparation.—Add to about 20 c.c. of dilute nitric acid, in a beaker glass, sufficient ammonia water to give it a distinct ammoniacal odor; filter, concentrate, keeping the ammonia in slight excess, and set aside in cool place for crystals to form:—



Properties.—These crystals contain twelve molecules of water of crystallization, which it is desirable to get rid of before using the salt. By exposure to a temperature of 155°C . the water gradually escapes, and the fused or granulated salt is ready to be converted into nitrogen monoxide (laughing gas), which takes place at about 185°C . according to the following reaction:—

AMMONIUM OXALATE, $(\text{NH}_4)_2\text{C}_2\text{O}_4$.

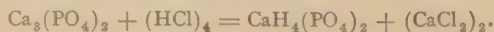
Preparation.—Dilute 20 c.c. of solution of ammonia with twice its bulk of water, add a solution of oxalic acid until neutral, concentrate slightly, filter and set aside to crystallize. The crystals may be collected on a filter, and another crop obtained by concentrating the “mother liquor”:—



Ammonium carbonate is sometimes used for combining with the oxalic acid, but the neutralization is not as easily effected, besides it is not desirable on the ground of economy.

CALCIUM PHOSPHATE, $\text{Ca}_3(\text{PO}_4)_2$.

Preparation.—Finely powdered bone ash is digested for a short time with diluted hydrochloric acid. The solution filtered, boiled, filtered again, if necessary. The filtrate is treated with ammonia until it smells strongly of it. Collect the precipitate on a filter, wash by pouring on warm water until the washings are tasteless, and dry at a low temperature. The resulting powder is calcium phosphate, which exists in the bone ash and is dissolved by hydrochloric acid, forming acid calcium phosphate, as follows:—

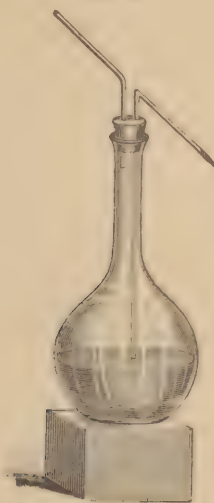


From this solution it is precipitated by ammonia, as follows:—



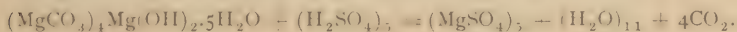
In addition to the ordinary apparatus with which a student supplies himself, there is required a *wash bottle* (Fig. 3), which it is well for every student to construct for himself, as it furnishes him valuable practice in cutting and bending glass tubing. This bottle is used in washing all precipitates, and is convenient as a water supply, which may be kept hot, if desired.

FIG. 3.



MAGNESIUM SULPHATE, $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$.

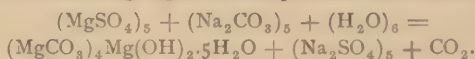
Preparation.—To about 5 c.c. of sulphuric acid, diluted with five or six times its volume of water, heated in a capsule, add powdered magnesium carbonate until effervescence ceases, and filter. Concentrate and set aside to crystallize.



MAGNESIUM CARBONATE, $(\text{MgCO}_3)_4\text{Mg}(\text{OH})_2 \cdot 5\text{H}_2\text{O}$.

Preparation.—On mixing solutions of magnesium sulphate and sodium carbonate and boiling, we get magnesium carbonate precipitated, while carbon dioxide escapes. The precipitate is very variable in its composition, depending on the

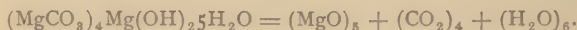
concentration of the solutions. When the U. S. P. product is obtained the following equation expresses the reaction:—



The precipitate washed with hot water and dried, serves for the following example of a compound prepared by *ignition*.

MAGNESIUM OXIDE, MgO .

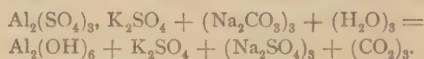
Preparation.—Heat some of the magnesium carbonate, prepared in the above reaction, in a porcelain crucible until, on taking out a small portion, placing in a test tube with a little water, heating to remove air bubbles, and adding a drop or two of hydrochloric acid, no effervescence is produced. This will require some time, and great care is necessary to determine when the powder fails to give an effervescence with the acid.



Zinc oxide may be prepared in a similar manner, from zinc carbonate. This differs from the magnesium oxide by being yellow while hot, and very pale yellow when cold.

ALUMINIUM HYDRATE, $\text{Al}_2(\text{OH})_6$.

Preparation.—To a solution of alum add a solution of sodium carbonate and boil. Allow the precipitate to settle, decant the clear supernatant liquid on a filter, add more hot water to the precipitate and again decant. Collect the precipitate on the filter, wash well with hot water and dry; the resulting white powder is *Aluminii Hydras*, U. S. P.



FERROUS SULPHATE, $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$.

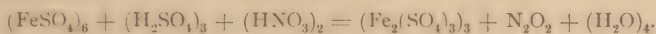
Preparation.—Add enough dilute sulphuric acid to some iron filings, or wire, in a beaker, to cover them. Allow the reaction to proceed, assisted by a little heat, until effervescence ceases. Filter from the excess of iron, concentrate, filter and crystallize.



These crystals should be rapidly dried and preserved in well stopped bottles, as they quickly become converted into ferric sulphate on exposure to air.

FERRIC SULPHATE, $\text{Fe}_2(\text{SO}_4)_3$.

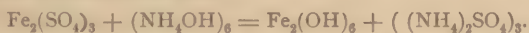
Preparation.—To a strong solution of ferrous sulphate add one-fourth its bulk of sulphuric acid, heat to the boiling point and drop in nitric acid as long as effervescence is produced and until the resulting liquid becomes of a clear reddish-brown color.



This is the Liquor Ferri Tersulphatis of the Pharmacopœia; and is the most convenient compound to use in the preparation of some of the other iron salts.

FERRIC HYDRATE, $\text{Fe}_2(\text{OH})_6$.

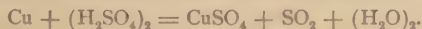
Preparation.—Dilute some of the above ferric sulphate solution with an equal bulk of water, add solution of ammonia until, after stirring, it smells strongly. The resulting precipitate is *ferric hydrate*, the well known antidote to arsenic. When needed for this purpose it is sufficient to pour the mixture on a muslin strainer, wash once or twice until the saline taste nearly disappears from the washings, when the compound is ready for use.



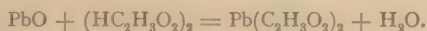
This preparation should always be freshly prepared when wanted for use as an antidote, as it loses H_2O on keeping, becoming a mixture of ferric oxide Fe_2O_3 and hydrate. This change takes place, although more slowly, when the compound is kept under water.

COPPER SULPHATE, $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$.

Preparation.—Boil copper turnings for some time, with strong sulphuric acid, in a fume closet, until the reaction ceases. Dilute with water, filter and crystallize.

LEAD ACETATE, $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 3\text{H}_2\text{O}$.

Preparation.—Lead oxide (litharge) is boiled with three or four times its weight of acetic acid, in a capsule, adding water from time to time, with more acid if necessary, until most of the oxide has disappeared. Filter, concentrate, keeping the solution acid, and set aside to crystallize:—



The solution or crystals should not be exposed to the fumes of the laboratory, for if there be only a small quantity of hydrogen sulphide in the room they will become black.

PART SECOND.

QUALITATIVE ANALYSIS.

PART II. QUALITATIVE ANALYSIS.

SECTION I.

BASES.

GROUP I.—POTASSIUM, SODIUM, LITHIUM, AMMONIUM.

REACTIONS OF POTASSIUM (K).

Use a solution of potassium chloride (KCl).

1. PtCl_4 causes a yellow crystalline precipitate of K_2PtCl_6 , soluble in excess of water. The delicacy of this reaction is increased by the addition of alcohol.

2. $\text{H}_2\text{C}_4\text{H}_4\text{O}_6$ in concentrated solution produces a white crystalline precipitate of **potassium acid tartrate**— $\text{KHC}_4\text{H}_4\text{O}_6$, soluble in excess of water, readily in hot water, acids or potassium hydrate. The addition of alcohol and violent agitation facilitate the formation of this precipitate.

3. A fragment of potassium salt on the loop of a platinum wire, held in the colorless flame of a Bunsen gas lamp imparts a violet color. This reaction is interfered with by the presence of sodium salts, which color the flame yellow, but the yellow rays may be destroyed by viewing the flame through blue glass.

4. Potassium salts are not volatile at a low red heat; at a white heat they are slowly volatilized.

REACTIONS OF SODIUM (Na).

Use a solution of sodium chloride (NaCl).

1. Sodium salts color the gas flame yellow; so delicate is this reaction that the merest traces are revealed by it.

2. The salts of sodium are not volatile at a low red heat, but slowly volatilize at a white heat.

REACTIONS OF LITHIUM (Li).

Use a solution of lithium chloride (LiCl).

1. Na_2HPO_4 added to a strong solution produces, on boiling, a white precipitate of **lithium phosphate**— Li_3PO_4 .

2. Lithium salts impart an intense crimson color to the gas flame. This is somewhat interfered with by sodium salts, but the yellow color may be excluded by blue glass, which if not too dark will allow the crimson rays to pass through. These must not be confused with the potassium rays, which will pass through a deep blue glass.

3. Lithium salts do not volatilize at a low red heat, but are slowly volatilized at a white heat.

REACTIONS OF AMMONIUM (NH_4).

Use a solution of ammonium chloride (NH_4Cl).

1. PtCl_4 produces, in strong solution, a yellow crystalline precipitate of **ammonium platino-chloride**— $(\text{NH}_4)_2\text{PtCl}_6$.

2. NaOH on heating causes the evolution of **ammonia**— NH_3 , detected by the odor, by holding near a glass rod moistened with HCl , which will produce dense white fumes of NH_4Cl , or by holding in the mouth of the tube a strip of moistened red litmus paper, when it will immediately become blue. Care must be taken, in this last test, to prevent any of the alkaline liquid coming in contact with the paper, as it would likewise cause the blue color.

3. $\text{H}_2\text{C}_4\text{H}_4\text{O}_6$ added to a concentrated solution produces a white precipitate of **ammonium acid tartrate**— $\text{NH}_4\text{HC}_4\text{H}_4\text{O}_6$, soluble in slight excess of water.

4. Ammonium salts are volatile at a low red heat.

SUMMARY OF TESTS WITH SOLUBLE SALTS OF GROUP I.

	K	Na	Li	NH_4
PtCl_4	Yellow Precipitate	No Precipitate	No Precipitate	Yellow Precipitate
$\text{H}_2\text{C}_4\text{H}_4\text{O}_6$	White Precipitate	No Precipitate	No Precipitate	White Precipitate
Flame	Violet	Yellow	Crimson	None
Volatility	Not Volatile	Not Volatile	Not Volatile	Volatile

DIRECTIONS FOR THE DETECTION OF THE
BASES IN A SOLUTION CONTAINING
SOLUBLE SALTS OF GROUP I.

To a small portion of the solution add NaOH and heat; NH_3 will be given off if ammonium salts are present, and may be detected by the odor, or by moistened red litmus paper.

Evaporate another portion of the solution to dryness, transfer to a porcelain crucible, and heat until the white fumes of ammonium salts cease to be given off. Dissolve the residue in a few drops of H_2O with a drop or two of HCl , and add PtCl_4 ; K, if present, will be precipitated.

A loop of platinum wire dipped in the original solution and held in the colorless gas flame will give evidence of Na and Li.

If Na be present in excess, so as to obscure the Li flame, evaporate a portion of the original solution to dryness, dissolve in the smallest possible amount of H_2O add Na_2HPO_4 and boil, filter off the Li_3PO_4 , wash with a little hot water containing NH_4OH , and dissolve in a few drops of HCl . With this solution Li may be detected by the flame test.

GROUP II.—BARIUM, STRONTIUM, CALCIUM,
MAGNESIUM.

REACTIONS OF BARIUM (Ba).

Use a solution of barium chloride (BaCl_2).

1. H_2SO_4 produces an immediate precipitate of **barium sulphate**— BaSO_4 , insoluble in boiling hydrochloric or nitric acid.
2. K_2CrO_4 even in dilute solutions causes a yellow precipitate of **barium chromate**— BaCrO_4 , soluble in hydrochloric or nitric acid, but insoluble in acetic acid.
3. $(\text{NH}_4)_2\text{CO}_3$ precipitates white **barium carbonate**— BaCO_3 , soluble in acetic acid.
4. $(\text{NH}_4)_2\text{HPO}_4$ produces a white precipitate of **barium phosphate**— BaHPO_4 , soluble in acetic and in hydrochloric acid.

5. $(\text{NH}_4)_2\text{C}_2\text{O}_4$ causes a white precipitate of **barium oxalate**— BaC_2O_4 , slightly soluble in acetic acid. This precipitation will not take place in very dilute solutions.

6. A loop of platinum wire moistened with the solution colors the gas flame green when held in it.

REACTIONS OF STRONTIUM (Sr).

Use a solution of strontium nitrate ($\text{Sr}(\text{NO}_3)_2$).

1. H_2SO_4 forms a white precipitate of **strontium sulphate**— SrSO_4 , immediately, if the solution be strong, but not until after some time, if it be very dilute.

2. K_2CrO_4 produces no precipitate in the presence of acetic acid, but if the solution be made alkaline with KOH , a yellow precipitate, **strontium chromate**— SrCrO_4 falls.

3. $(\text{NH}_4)_2\text{CO}_3$ produces a white precipitate of **strontium carbonate**— SrCO_3 , soluble in acetic and the stronger acids. Na_2CO_3 produces the same precipitate.

4. $(\text{NH}_4)_2\text{HPO}_4$ forms a white precipitate of **strontium phosphate**— SrHPO_4 , soluble in acids.

5. $(\text{NH}_4)_2\text{C}_2\text{O}_4$ causes the precipitation of white **strontium oxalate**— SrC_2O_4 , sparingly soluble in acetic acid, but readily soluble in HCl .

6. Strontium salts impart an intense red to the colorless gas flame.

REACTIONS OF CALCIUM (Ca).

Use a solution of calcium chloride (CaCl_2).

1. H_2SO_4 , in moderately dilute solutions, forms a white precipitate of **calcium sulphate**— CaSO_4 , soluble in excess of water.

2. $(\text{NH}_4)_2\text{CO}_3$ or Na_2CO_3 produces a white precipitate of **calcium carbonate**— CaCO_3 , soluble in acids. This precipitation is not complete unless the solution is boiled.

3. $(\text{NH}_4)_2\text{HPO}_4$ causes the precipitation of **calcium phosphate**— CaHPO_4 , soluble in acetic and the stronger acids.

4. $(\text{NH}_4)_2\text{C}_2\text{O}_4$ produces a white precipitate of **calcium oxalate**— CaC_2O_4 , insoluble in acetic acid, soluble in hydrochloric or nitric acid.

5. The salts of calcium color the flame yellowish-red.

REACTIONS OF MAGNESIUM (Mg).

Use a solution of magnesium sulphate (MgSO_4).

1. $(\text{NH}_4)_2\text{CO}_3$ forms a white precipitate of **magnesium-ammonium carbonate**— $\text{MgCO}_3(\text{NH}_4)_2\text{CO}_3$, soluble in NH_4Cl . By preceding the addition of the reagent, by that of NH_4Cl , a much smaller quantity will suffice to keep the precipitate in solution than will be required to dissolve it after once formed.

2. KOH , NaOH or NH_4OH produces a white precipitate of **magnesium hydrate**— $\text{Mg}(\text{OH})_2$, soluble in NH_4Cl .

3. $(\text{NH}_4)_2\text{HPO}_4$ with NH_4Cl and NH_4OH produces a white crystalline precipitate of **ammonium-magnesium phosphate**— $\text{Mg}(\text{NH}_4)\text{PO}_4$, slightly soluble in water, but almost insoluble in water containing NH_4OH . Violent agitation or stirring assists in the formation of this precipitate. $(\text{NH}_4)_2\text{HAsO}_4$ under similar circumstances precipitates white $\text{Mg}(\text{NH}_4)\text{AsO}_4$.

4. Magnesium salts impart no color to the flame.

SUMMARY OF TESTS WITH SOLUBLE SALTS OF GROUP II.

	Ba	Sr	Ca	Mg
H_2SO_4	White Precipitate insoluble in acids	White Precipitate insoluble in acids	White Precipitate soluble in excess of H_2O	No Precipitate
K_2CrO_4	Yellow Precipitate insoluble in acetic acid	No Precipitate unless alkaline	No Precipitate	No Precipitate
$(\text{NH}_4)_2\text{CO}_3$	White Precipitate	White Precipitate	White Precipitate	White Precipitate soluble in NH_4Cl
$(\text{NH}_4)_2\text{HPO}_4$	White Precipitate	White Precipitate	White Precipitate	White Precipitate
$(\text{NH}_4)_2\text{C}_2\text{O}_4$	White Precipitate in strong solution	White Precipitate in strong solution	White Precipitate in dilute solutions	No Precipitate unless concentrated
NH_4OH	No Precipitate	No Precipitate	No Precipitate	White Precipitate

DIRECTIONS FOR THE DETECTION OF THE BASES IN A SOLUTION CONTAINING SOLUBLE SALTS OF GROUP II.

Add NH_4Cl , NH_4OH and $(\text{NH}_4)_2\text{CO}_3$, boil and filter.

Ppt. Ba, Sr, Ca Wash, dissolve in HCl , H_2O_2 , add K_2CrO_4 , filter.		Filt. Mg Add $(\text{NH}_4)_2\text{HPO}_4$, agitate. White ppt. if Mg be present.	
Ppt. Ba	Filtrate Sr, Ca Add very dilute H_2SO_4 , allow to stand 10 minutes, filter.		
	Ppt. Sr Confirm by flame test	Filt. Ca Add $(\text{NH}_4)_2\text{C}_2\text{O}_4$ white ppt.	

DIRECTIONS FOR THE ANALYSIS OF A SOLUTION CONTAINING SOLUBLE SALTS OF ALL THE PRECEDING ELEMENTS.

Add NH_4Cl , NH_4OH and $(\text{NH}_4)_2\text{CO}_3$, boil and filter.

Ppt. Ba, Sr, Ca Wash, dissolve in HCl , add K_2CrO_4 , filter.		Filtrate Mg, K, Na, Li, NH_4 Add $(\text{NH}_4)_2\text{HPO}_4$, agitate, filter.	
Ppt. Ba Yellow	Filtrate Sr, Ca Add very dilute H_2SO_4 , allow to stand, filter.	Ppt. Mg	Filtrate K, Na, Li, NH_4 Evaporate to dryness, ignite, dissolve in a small quantity of H_2O , add Na_2HPO_4 , boil, filter.
	Ppt. Sr Confirm by flame test		Ppt. Li Confirm by flame test
	Filt. Ca Add $(\text{NH}_4)_2\text{C}_2\text{O}_4$, white ppt.		Filtrate K, Na, NH_4 Concentrate, add HCl and PtCl_4 , yellow ppt. = K Test for Na and NH_4 in original solution.

PRECAUTIONS TO BE OBSERVED IN THE PRECEDING CHARTS.

Ammonium chloride must be added in excess, in order to keep the magnesium salts in solution when the hydrate and carbonate are added. **Ammonium hydrate** is added until the liquid smells of it. **Ammonium carbonate** is added as long as a precipitate is produced. In order to determine this to a certainty, a portion of the filtrate is tested with a little more of the reagent, when, if no precipitate occurs, the analysis may be proceeded with. This precaution of applying more of the reagent to a portion of the filtrate, to prove the complete precipitation, should be exercised in every case, as it is important to add just sufficient of the reagent to accomplish the object, but always to avoid a large excess.

The analysis of the above solutions may be much simplified in many cases, by adding solution of CaSO_4 to the original solution. If a precipitate form immediately Ba is present, Sr and Ca may be. If a precipitate form after some time Ba is absent, Sr is present, and Ca may be present. If no precipitate be formed Ba and Sr are absent, and Ca may be tested for in another portion with $(\text{NH}_4)_2\text{C}_2\text{O}_4$. The above charts suppose all the elements to be present, in which case they afford the simplest means of detecting them.

GROUP III.—MANGANESE, ZINC, COBALT, NICKEL.

REACTIONS OF MANGANESE (Mn).

Use a solution of manganous sulphate (MnSO_4).

1. NH_4HS , in neutral or alkaline solution, precipitates the flesh-colored **manganous sulphide**— MnS , which on exposure to air becomes brown. HCl , HNO_3 and $\text{HC}_2\text{H}_3\text{O}_2$ dissolve this precipitate, but it is insoluble in alkalies. NH_4Cl facilitates the separation of the precipitate, while the salts of the organic acids and excess of NH_4OH prevent it.

2. KOH or NaOH produces a whitish precipitate of **Manganous hydrate**— $\text{Mn}(\text{OH})_2$, *insoluble* in excess.

3. NH_4OH likewise precipitates $\text{Mn}(\text{OH})_2$, *soluble* in excess. This precipitation is prevented by the previous addition of NH_4Cl .

4. $(\text{NH}_4)_2\text{CO}_3$ produces a white precipitate of **manganous carbonate**— MnCO_3 , *insoluble* in excess.

5. A fragment of a manganese salt fused on platinum foil with K_2CO_3 and KNO_3 will form a green mass containing **potassium manganate**— K_2MnO_4 , which on boiling with water forms a purple solution due to the formation of **potassium permanganate**— $\text{K}_2\text{Mn}_2\text{O}_8$.

6. A borax bead (formed by fusing on the loop of a platinum wire some borax until it becomes a clear glass) with manganese, in the oxidizing blowpipe flame, becomes violet while hot, and a fine amethyst color on cooling.

REACTIONS OF ZINC (Zn).

Use a solution of zinc sulphate (ZnSO_4).

1. NH_4HS produces a white precipitate of **zinc sulphide**— ZnS , insoluble in acetic acid, readily soluble in dilute hydrochloric acid.

2. NaOH and NH_4OH give white precipitates of **zincate**— $\text{Zn}(\text{OH})_2$, readily soluble in excess, forming *zincate* as $\text{Zn}(\text{OK})_2$. $\text{Zn}(\text{OH})_2$ is again precipitated on boiling.

3. $(\text{NH}_4)_2\text{CO}_3$ forms a white precipitate of **basic zinc carbonate**— $(\text{ZnCO}_3)_2(\text{Zn}(\text{OH})_2)_3$, readily *soluble* in excess.

4. K_2CO_3 or Na_2CO_3 produces a similar precipitate, *insoluble* in excess.

5. On charcoal, before the blowpipe, metallic zinc volatilizes and burns, forming an incrustation of oxide, which is yellow while hot, becoming white on cooling: if this coating be moistened with a drop of cobaltous nitrate, and again heated in the outer flame, it becomes *green*.

REACTIONS OF COBALT (Co).

Use a solution of cobaltous nitrate ($Co(NO_3)_2$).

1. NH_4HS , produces a black precipitate of **cobaltous sulphide**— CoS , insoluble in acetic acid, and cold dilute hydrochloric acid. The precipitation is promoted by the presence of NH_4Cl .

2. KOH or $NaOH$ produces a blue precipitate of **cobaltous hydrate**— $Co(OH)_2$, *insoluble* in excess, and becoming pink on boiling or exposure to air.

3. NH_4OH causes a similar precipitate of $Co(OH)_2$, *soluble* in excess with a red color. Sugar and some other organic compounds prevent the precipitations by the alkalies. The alkaline carbonates behave like their respective hydrates.

4. KCN gives a red-brown precipitate of **cobaltous cyanide**— $Co(CN)_2$, soluble in excess and reprecipitated by HCl ; if, however, the solution be boiled with only a few drops of HCl , the cobaltous cyanide will not be precipitated on the further addition of HCl , on account of the formation of **potassium cobalti-cyanide**— $K_6Co_2(CN)_{12}$. *This experiment should be performed in a fume closet, in order to avoid inhaling the fumes of hydrocyanic acid.*

5. Salts of cobalt color the borax bead *blue* before the blowpipe.

REACTIONS OF NICKEL (Ni).

Use a solution of nickelous sulphate ($NiSO_4$).

1. NH_4HS forms a black precipitate of **nickelous sulphide**— NiS , insoluble in acetic acid and cold dilute hydrochloric acid. The precipitation is promoted by the presence of NH_4Cl .

2. KOH or NaOH produces a green precipitate of **nickelous hydrate**— $\text{Ni}(\text{OH})_2$, *insoluble* in excess.

3. NH_4OH gives a similar precipitate, *soluble* in excess, with a blue color. Sugar and some other organic compounds prevent the precipitations by the alkalis. The alkaline carbonates behave like their respective hydrates.

4. KCN produces a yellowish-green precipitate of **nickelous cyanide**— $\text{Ni}(\text{CN})_2$, soluble in excess, and re-precipitated by HCl even after boiling, also precipitated, after adding HCl and boiling, by KOH. This is used as a method of distinguishing and separating nickel and cobalt, the latter *not* precipitating under these circumstances with KOH. *This experiment should be performed in a fume closet.*

5. The salts of nickel color the borax bead violet while hot, and reddish-brown when cold.

SUMMARY OF TESTS WITH SOLUBLE SALTS OF GROUP III.

	Mn	Zn	Co	Ni
NH_4HS	Flesh colored precipitate	White precipitate	Black precipitate	Black precipitate
KOH	White precipitate insoluble in excess	White precipitate soluble in excess	Blue precipitate insoluble in excess	Green precipitate insoluble in excess
NH_4OH	White precipitate soluble in excess	White precipitate soluble in excess	Blue precipitate soluble in excess	Green precipitate soluble in excess
$(\text{NH}_4)_2\text{CO}_3$	White precipitate insoluble in excess	White precipitate soluble in excess	Blue precipitate soluble in excess	Green precipitate soluble in excess

DIRECTIONS FOR THE DETECTION OF THE BASES IN A SOLUTION CONTAINING SOLUBLE SALTS OF GROUP III.

Mn, Zn, Co, Ni. Add $\text{HC}_2\text{H}_3\text{O}_2$ and H_2S until the liquid smells strongly; filter.

Precipitate Zn, Co, Ni Wash, dissolve in HCl and HNO_3 , add KOH in excess, filter.		Filtrate Mn Add NH_4OH and NH_4HS , ppt. pink turning brown.	
Precip. Co, Ni. Wash, dissolve in HCl, add KCN in excess and boil with HCl until all odor of HCN disappears, then add KOH in excess, filter.		Filt. Zn Add NH_4HS white ppt.	
Precip. Ni green	Filt. Co Evaporate to dryness and test with borax bead.		

DIRECTIONS FOR THE DETECTION OF THE BASES IN A SOLUTION CONTAINING SOLUBLE SALTS OF ALL THE ELEMENTS
PREVIOUSLY CONSIDERED.

<p>Mn, Zn, Co, Ni, * Ba, Sr, Ca, Mg, K, Na, Li, NH₄ Add NH₄Cl, NH₄OH and NH₄HS, warm gently, filter.</p>					
<p>Ppt. Mn, Zn, Co, Ni. Wash, dissolve in HCl and HNO₃, neutralize with HC₂H₃O₂, add H₂S, filter.</p>		<p>Filt. Mn Add NH₄OH and NH₄HS ppt. pink turn- ing brown.</p>		<p>Filtrate Ba, Sr, Ca, Mg, K, Na, Li, NH₄. Add (NH₄)₂CO₃, boil, filter.</p>	
<p>Ppt. Zn, Co, Ni. Wash, dissolve in HCl and HNO₃, Add KOH in excess, filter.</p>		<p>Ppt. Ba, Sr, Ca Wash, dissolve in HC₂H₃O₂, add K₂CrO₄, filter.</p>		<p>Filt. Mg, K, Na, Li, NH₄. Add (NH₄)₂HPO₄, agitate, filter.</p>	
<p>Ppt. Co, Ni. Wash, dissolve in HCl, add KCN in ex- cess, boil with HCl until all odor of HCN disappears, add KOH in excess, filter.</p>		<p>Filt. Zn Add NH₄HS white ppt.</p>		<p>Ppt. Mg Filt. K, Na, Li, NH₄. Evaporate to dryness, ignite, dissolve in a small quantity of H₂O, add Na₂HPO₄, boil, filter.</p>	
<p>Ppt. Ni green</p>		<p>Ppt. Sr Confirm by flame test.</p>		<p>Ppt. Li Confirm by flame test.</p>	
<p>Filt. Co Evaporate to dryness and test with borax bead.</p>		<p>Ppt. Ca Add (NH₄)₂C₂O₄ white ppt.</p>		<p>Filt. K, Na, NH₄. Concentrate, add HCl and PtCl₄, yellow ppt. = K test original solution for Na and NH₄.</p>	

* When Ni is present it may remain in solution in the NH₄HS, in which case the filtrate will be dark. To avoid this the mixture should be boiled after the addition of NH₄HS until the excess is driven off.

GROUP IV.—IRON, CERIUM, CHROMIUM, ALUMINIUM.

REACTIONS OF IRON *in ferrous state* (Fe^{II}).

Use a solution of ferrous sulphate (FeSO_4).

1. $\text{K}_4\text{Fe}(\text{CN})_6$ in a neutral or acid solution gives a white (rapidly changing to light blue) precipitate of **potassium ferrous ferrocyanide**— $\text{K}_2\text{Fe}_2(\text{CN})_6$, also known as Everett's salt. Alkalies decompose this precipitate, forming ferrous hydrate and a ferrocyanide of the base used.

2. $\text{K}_6\text{Fe}_2(\text{CN})_{12}$ in a neutral or slightly acid solution, forms a dark blue precipitate of **ferrous ferricyanide**— $\text{Fe}_3\text{Fe}_2(\text{CN})_{12}$, known as Turnbull's blue. If the solutions be very dilute there is produced merely a deep blue-green coloration.

3. KCNS produces no change.

4. H_2S in acid solution does not form a precipitate.

5. NH_4HS with a neutral or alkaline solution forms a black precipitate of **ferrous sulphide**, soluble in HCl or HNO_3 . NH_4Cl promotes the formation of this precipitate.

6. NH_4OH in the absence of NH_4Cl produces a dirty green precipitate of **ferrous hydrate**— $\text{Fe}(\text{OH})_2$. This precipitate rapidly becomes reddish-brown, owing to absorption of oxygen.

7. KOH produces a dirty green precipitate of **ferrous hydrate**— $\text{Fe}(\text{OH})_2$, similar to that produced by ammonia. Non-volatile organic substances, as sugar and some acids, retard the precipitations by NH_4OH and KOH .

8. Na_2CO_3 causes a white precipitate of **ferrous carbonate**— FeCO_3 , which rapidly becomes brown, from absorption of oxygen. This rapid oxidation is prevented by the use of distilled water and sugar.

REACTIONS OF IRON *in ferric state* (Fe_2^{VI}).

Use a solution of ferric chloride (Fe_2Cl_6).

1. $\text{K}_4\text{Fe}(\text{CN})_6$ in a neutral or acid solution produces a dark blue precipitate of **ferric ferrocyanide**— $(\text{Fe}_2)_2(\text{Fe}(\text{CN})_6)_3$, decomposed by alkalies.

2. $\text{K}_6\text{Fe}_2(\text{CN})_{12}$ forms no precipitate, but produces a deep reddish-brown color. The olive-green color sometimes produced in this reaction is due to traces of ferrous salt.

3. KCNS imparts to acid solutions a deep blood-red color, due to the formation of **ferric sulphocyanate**. This color is immediately destroyed by HgCl_2 . Dilute solutions show this reaction best.

4. H_2S forms in acid solutions a white turbidity due to separation of sulphur; the ferric salt being at the same time reduced to the ferrous condition. In alkaline solutions this reagent acts as an alkaline sulphide.

5. NH_4HS causes a black precipitate of **ferrous sulphide**— FeS , sulphur separating at the same time.

6. NH_4OH precipitates reddish-brown **ferric hydrate**— $\text{Fe}_2(\text{OH})_6$; non-volatile organic acids and sugar prevent this precipitation.

7. KOH and NaOH react like NH_4OH .

8. Na_2CO_3 and the other alkaline carbonates precipitate **ferric hydrate**— $\text{Fe}_2(\text{OH})_6$.

9. With borax in the oxidizing blowpipe flame, ferrous and ferric compounds give a dark yellow color to red beads while hot, and yellow when cold. In the reducing flame the beads change to bottle-green.

REACTIONS OF CERIUM (Ce).

Use a solution of cerous chloride (CeCl_2).

1. NH_4HS causes a white precipitate of **cerous hydrate**— $\text{Ce}(\text{OH})_2$.

2. NH_4OH produces the same white precipitate of $\text{Ce}(\text{OH})_2$, as also do KOH and NaOH .

3. $(\text{NH}_4)_2\text{C}_2\text{O}_4$ or $\text{H}_2\text{C}_2\text{O}_4$ forms a white precipitate of **cerous oxalate**— CeC_2O_4 . Organic matter does not interfere with the formation of this precipitate.

4. With borax bead before the blowpipe the salts of cerium behave like those of iron.

REACTIONS OF ALUMINIUM (Al).

Use a solution of alum ($\text{K}_2\text{SO}_4\text{Al}_2(\text{SO}_4)_3$).

1. NH_4HS produces a white precipitate of **aluminium hydrate**— $\text{Al}_2(\text{OH})_6$, while H_2S escapes.

2. NH_4OH forms a white precipitate of **aluminium hydrate**

— $\text{Al}_2(\text{OH})_6$, insoluble in excess; this is an important distinction from zinc.

3. KOH and NaOH produce a similar white precipitate of $\text{Al}_2(\text{OH})_6$, soluble in excess, forming aluminates of the base, as $\text{Al}_2(\text{OK})_6$. This is not reprecipitated by boiling (distinction from chromium).

4. Na_2CO_3 and the other alkaline carbonates precipitate white gelatinous aluminium hydrate— $\text{Al}_2(\text{OH})_6$, insoluble in excess, CO_2 escaping at the same time.

The presence of non-volatile organic acids and sugar prevent the complete precipitation in the above reactions.

REACTIONS OF CHROMIUM (Cr).

Use a solution of chromic chloride (Cr_2Cl_6).

1. NH_4HS produces a greenish precipitate of **chromic hydrate**— $\text{Cr}_2(\text{OH})_6$, H_2S escaping.

2. NH_4OH forms the same greenish precipitate of $\text{Cr}_2(\text{OH})_6$, insoluble in excess.

3. KOH and NaOH produce the same precipitate soluble in excess, but reprecipitated on boiling (distinction from aluminium).

4. Na_2CO_3 and the other alkaline carbonates, precipitate **green basic carbonates**.

5. KNO_3 and K_2CO_3 fused with chromium compounds become yellow from formation of **potassium chromate**— K_2CrO_4 . AgNO_3 and $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$ are important tests for this compound; the former precipitates red **silver chromate**, the latter yellow **lead chromate**.

6. With the borax bead in the inner blowpipe flame, chromium compounds give a green color.

SUMMARY OF TESTS WITH SOLUBLE SALTS OF GROUP IV.

	Fe(ous)	Fe(ic)	Ce	Al	Cr
$K_4Fe(CN)_6$	White ppt. turning blue	Deep blue precipitate			
$K_6Fe_2(CN)_{12}$	Deep blue precipitate	No ppt. brownish-red color			
KCNS	No change	Blood-red color			
NH_4HS	Black ppt.	Black ppt.	White ppt.	White ppt.	Greenish ppt.
NH_4OH	Dirty green ppt.	Reddish-brown ppt.	White ppt.	White ppt. insoluble in excess	Greenish ppt. insoluble in excess
KOH	Dirty green ppt.	Reddish-brown ppt.	White ppt.	White ppt. soluble in excess, <i>not</i> reprecipitated by boiling	Green ppt. soluble in excess reprecipitated by boiling
Na_2CO_3	White ppt. becoming dark	Reddish-brown ppt.	White ppt.	White ppt.	Green ppt.

DIRECTIONS FOR THE DETECTION OF THE BASES IN A SOLUTION CONTAINING SOLUBLE SALTS OF GROUP IV.

Evaporate a portion of the solution to dryness, fuse on platinum foil with Na_2CO_3 and KNO_3 , boil with water and filter.

Residue, Fe, Ce Dissolve in HCl, add $H_2C_4H_4O_6$ and NH_4OH in excess. Ppt. Ce white Test original solution for Fe(ous) and Fe(ic)	Filtrate Al, Cr Yellow if Cr be present, divide in two parts	
	Al Add NH_4Cl white gelatinous ppt.	Cr Add $Pb(C_2H_3O_2)_2$ yellow ppt.

PRECAUTIONS AND OBSERVATIONS ON THE PRECEDING CHART.

1. It is particularly desirable not to filter immediately after adding NH_4HS in the first precipitation, otherwise the Mn will not be thoroughly precipitated.

2. When the first precipitate is dissolved in HCl and HNO_3 , great care must be exercised to thoroughly oxidize the Fe by boiling with the HNO_3 , otherwise the precipitation by NH_4OH will not be complete.

3. The precipitate of aluminium hydrate is difficult to observe, as it floats in the solution, instead of falling to the bottom. Warming the solution will usually render it visible.

4. When phosphoric acid is present the members of Group II precipitate with Group IV, and require an entirely different method of separation. It is desirable, however, for the student first to familiarize himself with these simpler soluble salts, and undertake the more difficult cases of salts insoluble in water after the acids have been considered. See page 71.

GROUP V.—ARSENIC, ANTIMONY, TIN, GOLD, PLATINUM.

REACTIONS OF ARSENIC (As).

(a) ARSENIOS COMPOUNDS.

Use a solution of As_2O_3 in water.

1. H_2S passed into the solution produces a yellow color, but no precipitate until HCl is added, when a yellow precipitate of **arsenious sulphide**— As_2S_3 , falls. This precipitate is insoluble in strong HCl , but soluble in NH_4HS , NH_4OH and $(\text{NH}_4)_2\text{CO}_3$.

2. NH_4HS causes the formation of **arsenious sulphide**, which remains in solution as **ammonium sulpharsenite**— $(\text{NH}_4)_3\text{AsS}_3$. On the addition of HCl arsenious sulphide is precipitated.

3. AgNO_3 produces no precipitate until a few drops of dilute ammonia solution are added, when a yellow precipitate of **silver arsenite**— Ag_3AsO_3 , falls, soluble in HNO_3 and in NH_4OH .

4. CuSO_4 under similar circumstances produces a yellowish-green precipitate of **cupric arsenite**— CuHAsO_3 .

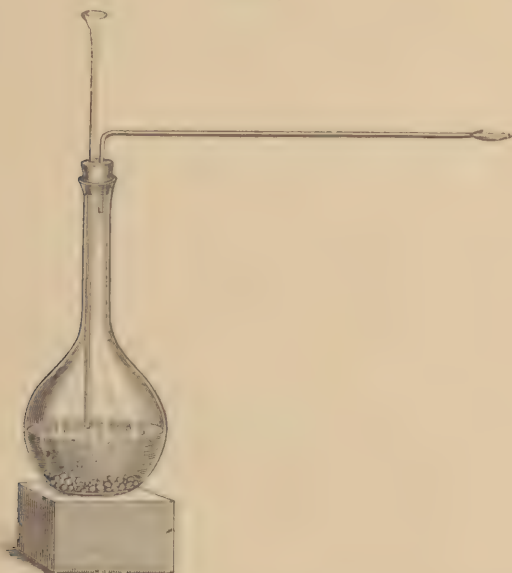
(b) ARSENIC COMPOUNDS.

Use a solution of sodium arsenate (Na_2HAsO_4).

1. H_2S causes in acid solution only a yellow precipitate of **arsenious sulphide**— As_2S_3 , mixed with sulphur. This reaction takes place slowly, but is accelerated by heat.

2. NH_4HS produces no precipitate, but forms **arsenic sulphide**— As_2S_5 , which remains in solution as **ammonium sulpharsenate**— $(\text{NH}_4)_3\text{AsS}_4$. Upon the addition of HCl As_2S_5 is precipitated, and not As_2S_3 and S .

FIG. 4.



3. AgNO_3 with a small amount NH_4OH produces a chocolate colored precipitate of **silver arsenate**— Ag_3AsO_4 , soluble in HNO_3 and NH_4OH .

4. CuSO_4 under similar circumstances forms a bluish-green precipitate of **cupric arsenate**— CuHAsO_4 .

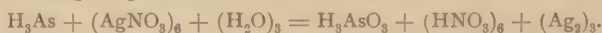
The following tests are applicable to both arsenious and arsenic compounds.

1. *Marsh's Test*.—Generate hydrogen in the usual way, allowing it to escape through a glass tube drawn out at the end so as to form a small orifice (Fig. 4). In very exact cases

the gas should be dried by passing over calcium chloride. When all the air has been expelled (which should be determined by collecting a small test tube full and holding its mouth to a flame, if the gas burn quietly, without explosion, it is pure) ignite the escaping gas; it should burn with a colorless or yellow flame; in the latter case it is due to the sodium in the glass. A piece of cold porcelain, a small crucible lid is best, is pressed down on the flame; there should be no deposit on it. Add now through the funnel tube a solution of arsenic, washing it down with a little water. The flame will become of a pale blue color, due to the formation of **hydrogen arsenide**— H_3As . On bringing the crucible lid into the flame now, a blackish-brown deposit with metallic lustre will form on it. This deposit is readily soluble in a solution of sodium or calcium hypochlorite.

2. *Reinsch's Test*.—Boil some strips of copper with dilute HCl ; if no discoloration of the copper takes place, the arsenic solution may be added. The copper immediately becomes coated with an iron-gray metallic film. Pour off the liquid, dry the copper by holding it in the lamp with the fingers so it may not become too hot, place in a clean, dry, narrow test tube, and heat gently, when a white ring of As_2O_3 will form on the tube above the copper, readily distinguished by the characteristic octahedral shape of the crystals.

Fleitman's Test.—Generate hydrogen in a test tube with zinc and solution of potassium hydrate; moisten a piece of filter paper with one drop of solution of silver nitrate, place it over the mouth of the tube and heat; there should be no coloration of the spot on the paper. Now add some compound of arsenic; the silver nitrate will immediately become black, owing to production of metallic silver.



Before the blowpipe, on charcoal, arsenic volatilizes, with the characteristic odor of garlic.

REACTIONS OF ANTIMONY (Sb).

Use a solution of tartar emetic ($\text{KSbOC}_4\text{H}_4\text{O}_6$), acidified with HCl .

1. H_2S forms an orange precipitate of **antimonous sulphide**— Sb_2S_3 , soluble in NH_4HS , and in concentrated HCl , but insoluble in $(\text{NH}_4)_2\text{CO}_3$.

2. NH_4HS produces an orange precipitate of **antimonous sulphide**, readily soluble in excess, forming **ammonium sulph-antimonite**— $(\text{NH}_4)_3\text{SbS}_3$, from which HCl again precipitates Sb_2S_3 .

3. KOH or NaOH precipitates white, bulky **antimonous hydrate**— $\text{Sb}(\text{OH})_3$, soluble in excess.

4. NH_4OH precipitates the same compound insoluble in excess.

5. *Marsh's Test* gives the same result as with arsenic; the black spot, however, is *insoluble* in sodium or calcium hypochlorite solution, but *soluble* in NH_4HS .

6. *Reinsch's Test* causes a deposit on copper, as with arsenic, but when heated in a tube there is formed a white amorphous ring, which is readily distinguished from the crystalline one of arsenic.

7. *Fleitman's Test* gives no result with antimony compounds.

8. On charcoal, with Na_2CO_3 , before the blowpipe, a metallic globule of antimony is produced, while characteristic fumes of the oxide are given off.

REACTIONS OF TIN (Sn).

(a) STANNOUS COMPOUNDS.

Use a solution of stannous chloride (SnCl_2).

1. H_2S precipitates dark brown **stannous sulphide**— SnS , soluble in concentrated HCl and in $(\text{NH}_4)_2\text{S}$, insoluble in NH_4HS .

2. KOH or NaOH precipitates white **stannous hydrate**— $\text{Sn}(\text{OH})_2$, *soluble* in excess; on boiling this solution SnO precipitates.

3. NH_4OH precipitates the same compound *insoluble* in excess.

4. HgCl_2 causes a white precipitate of Hg_2Cl_2 , converting the SnCl_2 into SnCl_4 .

(b) STANNIC COMPOUNDS.

Use a solution of stannic chloride (SnCl_4).

1. H_2S produces a yellow precipitate of **stannic sulphide**— SnS_2 , soluble in NH_4HS and in concentrated HCl .

2. KOH or NaOH precipitates white **stannic acid**— H_2SnO_3 , *soluble* in excess; on boiling no reprecipitation takes place—*distinction from stannous salts*.

3. NH_4OH produces the same precipitate *insoluble* in excess.

4. Heated on charcoal, before the blowpipe, with Na_2CO_3 , metallic tin is formed, with the production of a white incrustation of the oxide.

REACTIONS OF GOLD (Au).

Use a solution of auric chloride (AuCl_3).

1. H_2S precipitates black **auric sulphide**— Au_2S_3 , insoluble in HCl , soluble in $(\text{NH}_4)_2\text{S}$.

2. $\text{H}_2\text{C}_2\text{O}_4$ or FeSO_4 precipitates metallic gold as a finely divided brown powder.

3. SnCl_2 mixed with SnCl_4 (prepared by SnCl_2 and chlorine water) produces a purple-red precipitate or coloration (Purple of Cassius), consisting of the mixed oxides of gold and tin.

4. Heated on charcoal, before the blowpipe, metallic gold is produced.

REACTIONS OF PLATINUM (Pt).

Use a solution of platinum chloride (PtCl_4).

1. H_2S causes a brown precipitate of **platinic sulphide**— PtS_2 , insoluble in HCl , soluble in $(\text{NH}_4)_2\text{S}$.

2. KCl produces a yellow crystalline precipitate of **potassium platinic chloride**— K_2PtCl_6 .

3. NH_4Cl forms a similar precipitate of $(\text{NH}_4)_2\text{PtCl}_6$. The precipitation in both cases is facilitated by the addition of alcohol.

4. Zn, Fe and some other metals precipitate metallic platinum.

5. Heating on charcoal, before the blowpipe, produces the metal.

SUMMARY OF TESTS WITH SOLUBLE SALTS OF GROUP V.

	As	Sb	Sn	Au	Pt
H ₂ S	Yellow ppt. soluble in NH ₄ HS	Orange-red ppt. soluble in NH ₄ HS	Brown or Yellow ppt. soluble in (NH ₄) ₂ S	Black ppt. soluble in (NH ₄) ₂ S	Brown ppt. soluble in (NH ₄) ₂ S
KOH	No change	White ppt. soluble in excess	White ppt. soluble in excess	No change	With excess of HCl yellow ppt.
NH ₄ OH	No change	White ppt. insoluble in excess	White ppt. insoluble in excess	Red ppt. fulminating gold	With excess of HCl yellow ppt.
Marsh's Test	Black spot with metallic lustre, soluble in Ca(OCl) ₂	Black, sooty spot, soluble in (NH ₄)HS, insoluble in Ca(OCl) ₂			
Reinsch's Test	White sublimate of octahedral crystals	White amorphous sublimate			
Fleitman's Test	Dark spot of metallic silver	No change			

DIRECTIONS FOR THE DETECTION OF THE BASES IN A SOLUTION CONTAINING SOLUBLE SALTS OF GROUP V.

Add HCl and H₂S, collect the precipitate, transfer to a dish, boil with concentrated HCl; filter.

<p>Ppt. As, Au, Pt. If yellow, As only is present, if dark, wash, digest with (NH₄)₂CO₃, filter.</p>		<p>Filt. As. Acidify with HCl. Yellow ppt.</p>
<p>Ppt. Au, Pt. Dissolve in HCl and HNO₃. Divide in two parts.</p>		
<p>Au. Add SnCl₂ purple. Confirm by testing original solution.</p>	<p>Pt. Add KCl, yellow ppt. Confirm by testing original solution</p>	

Filt. Sb, Sn.
Dilute with H₂O, boil in a dish with a strip of
platinum foil, and a small piece of zinc so the
metals touch. The platinum will be coated
with black Sb, while the Sn will be de-
posited as a black sediment. This is dis-
solved in strong HCl and tested with
H₂Cl₂, white ppt. if Sn be present.
Treat the foil with a few drops of
HNO₃, dissolve in solution
H₂C₄H₄O₆, add H₂S,
orange ppt. if Sb
be present.

DIRECTIONS FOR THE DETECTION OF THE BASES IN A SOLUTION CONTAINING SOLUBLE SALTS
OF ALL THE ELEMENTS HERETOFORE CONSIDERED.

Ppt. As, Sb, Sn, Au, Pt. Wash, boil with concentrated HCl, filter.	As, Sb, Sn, Au, Pt, Fe, Ce, Al, Cr, Mn, Zn, Co, Ni, Ba, Sr, Ca, Mg, K, Na, Li, NH ₄ . Add HCl and H ₂ S until, after warming, the liquid smells strongly. filter.				
Ppt. As, Sb, Sn. Separate by the preceding Chart for Group V.	Filt. Fe, Ce, Al, Cr, Mn, Zn, Co, Ni, Ba, Sr, Ca, Mg, K, Na, Li, NH ₄ . Add NH ₄ Cl in excess, NH ₄ OH until after agitation its odor is perceptible, and NH ₄ HS, warm, filter.				
Ppt. Fe, Ce, Al, Cr. Wash, dry, fuse on foil with Na ₂ CO ₃ and KNO ₃ , boil with H ₂ O, filter.	Ppt. Fe, Ce, Al, Cr, Mn, Zn, Co, Ni. Wash, dissolve in HCl and HNO ₃ , add NH ₄ OH in excess, filter.				
Ppt. Fe, Ce. Wash, dissolve in HCl, add H ₂ C ₄ H ₄ O ₆ and NH ₄ OH in excess. White ppt. Test the original solution for Fe(ous) and Fe(ic).	Ppt. Zn, Co, Ni. Wash, dissolve in HCl and HNO ₃ , add excess of KOH, filter.				
Ppt. Fe, Ce. Wash, dissolve in HCl, add H ₂ C ₄ H ₄ O ₆ and NH ₄ OH in excess. White ppt. Test the original solution for Fe(ous) and Fe(ic).	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Al, Cr. Yellow if Cr be present, divide in two parts.				
Ppt. Fe, Ce. Wash, dissolve in HCl, add H ₂ C ₄ H ₄ O ₆ and NH ₄ OH in excess. White ppt. Test the original solution for Fe(ous) and Fe(ic).	Ppt. Co, Ni. Wash, dissolve in HCl, add KCN in excess, boil with HCl until all odor of HCN disappears, add KOH in excess, filter.				
Ppt. Fe, Ce. Wash, dissolve in HCl, add H ₂ C ₄ H ₄ O ₆ and NH ₄ OH in excess. White ppt. Test the original solution for Fe(ous) and Fe(ic).	Filt. Zn. Add NH ₄ HS white ppt.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.				
Ppt. Fe, Ce, Al, Cr. Yellow if Cr be present, divide in two parts.	Filt. Mn, Zn, Co, Ni. Acidify with HC _{2</}				

PRECAUTIONS AND OBSERVATIONS ON THE PRECEDING CHART.

A great deal of time may be saved by carefully noting the color of the precipitate produced by H_2S . If yellow, As only is present, if orange, Sb is present and As may be; when such a precipitate is obtained the readiest method of separation is to wash, and add to the precipitate $(\text{NH}_4)_2\text{CO}_3$. As will be dissolved and may be detected in the filtrate by adding HCl , while the Sb remains on the filter. When the precipitate is dark Au and Pt should be sought for in the original solution, as well as separated by the chart.

Only a small piece of zinc is necessary to effect the separation of Sn and Sb.

GROUP VI.—MERCURY(II), BISMUTH, COPPER, CADMIUM.

REACTIONS OF MERCURY *as mercuric salt* (Hg(II)).

Use a solution of mercuric chloride (HgCl_2).

1. H_2S or NH_4HS produces, when in small proportion, a whitish precipitate of $(\text{HgS})_2\text{HgCl}_2$; a further addition of the reagent, together with the application of heat causes the formation of a black precipitate of **mercuric sulphide**— HgS , insoluble in either HCl or HNO_3 , but soluble in a mixture of the two.

2. KOH or NaOH produces a yellow precipitate of **mercuric oxide**— HgO ; unless the reagent be in excess a brown precipitate of a basic salt is formed.

3. NH_4OH precipitates white **mercur-ammonium chloride**— NH_2HgCl .

4. K_2CrO_4 produces a red precipitate of **mercuric chromate**— HgCrO_4 .

5. KI precipitates **mercuric iodide**— HgI_2 , first yellow, but rapidly becoming scarlet. This precipitate is readily soluble in excess of KI or HgCl_2 .

6. SnCl_2 , in small quantity, in the presence of HCl , precipitates **mercurous chloride**— Hg_2Cl_2 . On the addition of a larger quantity of the reagent, the mercurous chloride is reduced to the metal, which may be collected into a globule.

7. Na_2CO_3 produces a reddish-brown precipitate of basic carbonate $\text{HgCO}_3(\text{HgO})_3$.

8. Before the blowpipe, HgO breaks up into Hg and O . HgS under similar circumstances sublimes unchanged.

REACTIONS OF BISMUTH (Bi).

Use a solution of bismuth nitrate ($\text{Bi}(\text{NO}_3)_3$).

1. H_2S or NH_4HS produces a black precipitate of **bismuth trisulphide**— Bi_2S_3 , insoluble in dilute acids and alkalies, soluble in boiling HNO_3 .

2. KOH , NaOH or NH_4OH forms a white precipitate of **bismuth hydrate**— $\text{Bi}(\text{OH})_3$, converted by boiling into the yellow **oxide**— Bi_2O_3 .

3. K_2CrO_4 precipitates yellow **bismuth chromate**— $\text{Bi}_2(\text{CrO}_4)_3$.

4. KI forms a brown precipitate of **bismuth iodide**— BiI_3 , soluble in excess of the reagent.

5. Na_2CO_3 precipitates white **oxycarbonate of bismuth**— $(\text{BiO})_2\text{CO}_3 \cdot \text{H}_2\text{O}$.

6. H_2O in excess, when there is not an excess of free acid, precipitates **bismuth subnitrate**— $\text{BiONO}_3 \cdot \text{H}_2\text{O}$. When the chloride is so diluted the **oxychloride**— BiOCl , separates.

7. Bismuth on charcoal, before the blowpipe, forms a hard bead of metal, with a characteristic incrustation of oxide—deep orange-yellow while hot, pale when cold.

REACTIONS OF COPPER (Cu).

Use a solution cupric sulphate (CuSO_4).

1. H_2S and NH_4HS precipitate black **cupric sulphide**— CuS , insoluble in dilute acids and alkalies, slightly soluble in NH_4HS , and entirely dissolved by boiling HNO_3 . This precipitation is prevented by KCN .

2. KOH or NaOH produces a light blue precipitate of **cupric hydrate**— $\text{Cu}(\text{OH})_2$, insoluble in excess, and converted by boiling into black **cupric oxyhydrate**— $(\text{CuO})_2\text{Cu}(\text{OH})_2$. In the presence of non-volatile organic acids this precipitation does not take place, but a blue color results.

3. NH_4OH in small quantity forms a greenish-blue precipitate, readily soluble in excess forming **tetra-ammonio-cupric sulphate**— $(\text{NH}_3)_4\text{CuSO}_4 \cdot \text{H}_2\text{O}$.

4. $\text{K}_4\text{Fe}(\text{CN})_6$ precipitates reddish-brown **cupric ferrocyanide**— $\text{Cu}_2\text{Fe}(\text{CN})_6$.

5. Metallic Fe or Zn precipitates red metallic Cu.

6. In the outer blowpipe flame copper salts color the borax bead green while hot, blue when cold. In the inner flame, after moistening with SnCl_2 , it becomes red, owing to formation of Cu_2O .

REACTIONS OF CADMIUM (Cd).

Use a solution of cadmium sulphate (CdSO_4).

1. H_2S or NH_4HS precipitates yellow **cadmium sulphide**— CdS , soluble in hot HNO_3 , but insoluble in NH_4HS or KCN .

2. KOH or NaOH produces a white precipitate of **cadmium hydrate**— $\text{Cd}(\text{OH})_2$, *insoluble in excess*.

3. NH_4OH causes the same white precipitate of $\text{Cd}(\text{OH})_2$, *soluble in excess*.

4. Na_2CO_3 produces a white precipitate of **cadmium carbonate**— CdCO_3 , *insoluble in excess*, but slightly soluble in ammonium salts, entirely soluble in NH_4OH .

5. On charcoal, before the blowpipe, the salts of cadmium are reduced to metal and volatilize, forming a brownish incrustation of oxide.

SUMMARY OF TESTS WITH SOLUBLE SALTS OF GROUP VI.

	Hg(ic)	Bi	Cu	Cd
H_2S or NH_4HS	Black ppt. insoluble in HNO_3	Black ppt.	Black ppt. soluble in KCN	Yellow ppt. insoluble in KCN
KOH	Yellow ppt.	White ppt.	Blue ppt.	White ppt.
NH_4OH	White ppt.	White ppt.	Blue ppt. soluble in excess	White ppt. soluble in excess
Na_2CO_3	Reddish-brown ppt.	White ppt.	Blue ppt.	White ppt.

DIRECTIONS FOR THE DETECTION OF THE BASES IN A SOLUTION CONTAINING SOLUBLE SALTS OF GROUP VI.

Add HCl and H_2S , collect, boil with HNO_3 ; filter.

Ppt. Hg.	Filt. Bi, Cu, Cd. Add NH_4OH in excess; filter.		
	Ppt. Bi.	Filt. Cu, Cd. Add H_2S , collect and wash; boil with dilute H_2SO_4 ; filter.	
		Ppt. Cu	Filt. Cd. Dilute, add H_2S , yellow ppt.

PRECAUTIONS AND OBSERVATIONS ON THE PRECEDING CHART.

1. Unless excess of H_2S be used, and the solution warmed, Hg will not be thoroughly precipitated.

2. When Sn, Au and Pt are present, the yellow ammonium sulphide $(NH_4)_2S$ must be used to dissolve Group V. When Cu is present some of it may be dissolved, so that any dark precipitate in that group should be tested for Cu.

3. When the precipitate insoluble in NH_4HS is boiled with HNO_3 , a black mass of sulphur is sometimes obtained. This is readily distinguished from Hg, because it floats as one mass on the liquid, while the mercury collects at the bottom as a heavy, black precipitate.

GROUP VII.—SILVER, MERCURY(OUS), LEAD.

REACTIONS OF SILVER (Ag).

Use a solution of silver nitrate ($AgNO_3$).

1. HCl or soluble chlorides precipitate white, curdy **silver chloride**— $AgCl$, insoluble in HNO_3 , soluble in NH_4OH , forming **ammonio-silver chloride**— $(AgCl)_2(NH_3)_3$, from which the chloride is again precipitated by acids.

2. H_2S or NH_4HS produces a black precipitate of **silver sulphide**— Ag_2S , insoluble in dilute acids and in alkalis, soluble in boiling HNO_3 .

3. KOH or $NaOH$ forms a grayish-brown precipitate of **siver oxide**— Ag_2O , insoluble in excess, but soluble in NH_4OH .

4. NH_4OH in small quantity, precipitates **silver oxide**, *soluble in excess*.

5. K_2CrO_4 produces a red precipitate of **silver chromate**— Ag_2CrO_4 , soluble in concentrated HNO_3 and in NH_4OH .

6. KI and KBr produce precipitates of **silver iodide**— AgI , yellow, insoluble in NH_4OH , and **silver bromide**— $AgBr$, yellowish-white, slowly soluble in NH_4OH .

7. KCN precipitates white **silver cyanide**— $AgCN$, soluble in excess and in HNO_3 .

8. Heated with Na_2CO_3 on charcoal, before the blowpipe, compounds of silver form a bright, metallic button, soluble in HNO_3 .

REACTIONS OF MERCURY, *as mercurous salt* ($\text{Hg}(\text{ous})$).*Use a solution of mercurous nitrate* ($\text{Hg}_2(\text{NO}_3)_2$).

1. HCl or soluble chlorides precipitate **mercurous chloride**— Hg_2Cl_2 , converted by strong HNO_3 into a mixture of HgCl_2 and $\text{Hg}(\text{NO}_3)_2$, also becoming black on the addition of NH_4OH , forming $\text{NH}_2\text{Hg}_2\text{Cl}$.
2. H_2S or NH_4HS precipitates a mixture of Hg with HgS .
3. KOH or NaOH produces a black precipitate of **mercurous oxide**— Hg_2O , insoluble in excess.
4. NH_4OH causes a black precipitate of **mercurous-ammonium nitrate**— $\text{NH}_2\text{Hg}_2\text{NO}_3$.
5. K_2CrO_4 forms an orange precipitate of **mercurous chromate**— Hg_2CrO_4 .
6. KI precipitates green **mercurous iodide**— Hg_2I_2 .
7. Before the blowpipe, mercurous salts volatilize, some being converted into mercuric salt and mercury, both of which sublime.

REACTIONS OF LEAD (Pb).*Use a solution of lead acetate* ($\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$).

1. HCl , or soluble chlorides, produce a white precipitate of **lead chloride**— PbCl_2 , soluble in hot water.
2. H_2S or NH_4HS precipitates black **lead sulphide**— PbS , insoluble in HCl , soluble in hot HNO_3 .
3. KOH or NaOH produces a white precipitate of **lead hydrate**— $\text{Pb}(\text{OH})_2$, soluble in large excess, forming **potassium or sodium plumbate**— K_2PbO_2 or Na_2PbO_2 .
4. NH_4OH precipitates white **basic lead hydrate**.
5. K_2CrO_4 produces a yellow precipitate of **lead chromate**— PbCrO_4 , soluble in KOH and in strong HNO_3 .
6. KI forms a yellow precipitate of **lead iodide**— PbI_2 , soluble in boiling water.
7. H_2SO_4 produces a white precipitate of **lead sulphate**— PbSO_4 , insoluble in acids, but soluble in solution of ammonium acetate or tartrate.
8. Na_2CO_3 precipitates white **basic lead carbonate**— $(\text{PbCO}_3)_2\text{Pb}(\text{OH})_2$.

9. Before the blowpipe, on charcoal, lead compounds are converted into a malleable globule of the metal, with the formation of some yellow oxide.

SUMMARY OF TESTS WITH SOLUBLE SALTS OF GROUP VII.

	Ag	Hg(ous)	Pb
HCl	White ppt. soluble in NH_4OH	White ppt., turning black with NH_4OH	White ppt. soluble in hot H_2O
H_2S and NH_4HS	Black ppt.	Black ppt.	Black ppt.
KOH	Brown ppt.	Black ppt.	White ppt.
NH_4OH	Brown ppt.	Black ppt.	White ppt.
Na_2CO_3	Brown ppt.	Black ppt.	White ppt.
K_2CrO_4	Red ppt.	Orange ppt.	Yellow ppt.
KI	Yellow ppt.	Green ppt.	Yellow ppt.

DIRECTIONS FOR THE DETECTION OF THE BASES IN A SOLUTION CONTAINING SOLUBLE SALTS OF GROUP VII.

Add HCl, collect, wash and pour on the filter boiling H_2O .

Ppt. Ag, Hg(ous). Pour on the filter NH_4OH .		Filt. Pb. Add H_2SO_4 white ppt.
Ppt. Hg(ous) black	Filt. Ag. Add HNO_3 in excess, white ppt.	

DIRECTIONS FOR THE DETECTION OF THE BASES IN A SOLUTION CONTAINING SOLUBLE SALTS
OF ALL THE ELEMENTS HERETOFORE CONSIDERED.

Ppt. Ag, Hg(ous), Pb. Wash, pour boiling H ₂ O on the filter.	Ag, Hg(ous), Pb, Hg(ic), Bi, Cu, Cd, As, Sb, Sn, Au, Pt, Fe, Ce, Al, Cr, Mn, Zn, Co, Ni, Ba, Sr, Ca, Mg, K, Na, Li, NH ₄ . Add HCl, filter.										
Ppt. Ag, Hg(ous), Pour on the filter NH ₄ OH.	Filt. Pb add H ₂ SO ₄ , white ppt.	Filt. Hg(ic), Bi, Pb, Cu, Cd, As, Sb, Sn, Au, Pt. Wash, digest with NH ₄ HS, filter.									
Ppt. Hg.	Ppt. Hg, Bi, Pb, Cu, Cd. Wash, boil with HNO ₃ , filter.	Filt. As, Sb, Sn, Au, Pt. Acidify with dilute HCl. Collect and wash the pre- cipitate (throw away the pre- filtrate), boil the ppt. with concentrated HCl, filter.	Ppt. Fe, Ce, Al, Cr. Wash, dry, fuse on foil with Na ₂ CO ₃ and KNO ₃ , boil with H ₂ O, filter.	Filt. Mn, Zn, Co, Ni. Acidify with HC ₂ H ₃ O ₂ , add H ₂ S, filter.	Ppt. Ba, Sr, Ca. Wash, dissolve in HC ₂ H ₃ O ₂ , add K ₂ CrO ₄ , filter.	Filt. Mg, K, Na, Li, NH ₄ . Add (NH ₄) ₂ HPO ₄ , agitate, filter.					
Ppt. Hg, Bi, Pb, Cu, Cd. Add NH ₄ OH in excess, filter.	Ppt. Bi. white.	Filt. Al, Cr. Yellow if Cr be present. Divide in two parts.	Ppt. Fe, Ce. Wash, dissolve in HCl, add H ₂ C ₂ O ₄ , add NH ₄ OH in excess, filter.	Filt. Mn. Add H ₂ SO ₄ , allow to stand, filter.	Ppt. Ba, Sr, Ca. Add very dilute H ₂ SO ₄ , allow to stand, filter.	Ppt. Filt. K, Na, Li, NH ₄ . Evaporate to dryness, ignite, dissolve in solution of Na ₂ HPO ₄ , filter.					
Ppt. Cu, Cd. Collect, boil the ppt. with dilute H ₂ SO ₄ , filter.	Ppt. Fe. Test original solution for Fe(ous) and Fe(c).	Al add white ppt.	Cr add white ppt.	Filt. Zn add NH ₄ HS white ppt.	Ppt. Sr. Con- firm white ppt.	Ppt. Filt. Li, K, Na, Con- firm NH ₄ Cl, by flame test.					
Ppt. Cd. Dilute, add H ₂ S, yellow ppt.	Ppt. Ni. Evapo- rate to dryness and test with borax bead.						Ppt. Filt. Li, K, Na, Con- firm NH ₄ Cl, by flame test.				

SPECIAL PRECAUTIONS TO BE OBSERVED IN THE EXAMINATION FOR BASES.

1. HCl may precipitate basic salts of Bi and Sb; these are readily distinguished from the bases of Group VII by dissolving on the further addition of HCl.

2. The precipitation of HCl should be performed in the cold, in order to prevent the PbCl_2 from dissolving.

3. Before commencing the analysis of a solution its action on litmus paper should always be noted. If alkaline a precipitate may be caused by HCl, on account of the presence of substances soluble in alkalies, as As_2S_3 in $(\text{NH}_4)_2\text{CO}_3$, and AgCl in NH_4OH . Or silicic acid from alkaline silicates may be precipitated, in which case it is necessary to precede the addition of HCl, by evaporation to dryness with HNO_3 , filtering out the insoluble silica, and proceeding with the filtrate in the usual way.

4. The addition of H_2S should be slow and interrupted from time to time, to warm and agitate the solution, and so continued until it smells strongly of the reagent.

SECTION II.

ACIDS.

REACTIONS OF HYDROCHLORIC ACID (HCl).

Use a solution of potassium chloride (KCl).

1. AgNO_3 produces a white, curdy precipitate of **silver chloride**— AgCl , insoluble in HNO_3 , readily soluble in NH_4OH .

2. $\text{Hg}_2(\text{NO}_3)_2$ causes a white precipitate of **mercurous chloride**— Hg_2Cl_2 , insoluble in HNO_3 , blackening on the addition of NH_4OH .

3. $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$ forms a white crystalline precipitate of **lead chloride**— PbCl_2 , soluble in 33 parts of boiling water.

4. Warmed with H_2SO_4 and MnO_2 causes the evolution of **Chlorine**, recognized by its odor and color.

5. On warming with H_2SO_4 **hydrochloric acid** is given off, recognized by its odor and intensely acid reaction; also by the dense white fumes of NH_4Cl produced by holding a rod moistened with NH_4OH near the mouth of the tube.

HYPOCHLOROUS ACID (HClO).

This acid is known in combination with calcium $\text{Ca}(\text{ClO})_2$, bleaching powder, and with sodium NaClO , Labarraque's solution. The chlorine-like odor developed on the addition of an acid is sufficient evidence of its presence.

CHLORIC ACID (HClO_3).

The compounds of potassium and sodium with this acid are best known.

1. H_2SO_4 added to one of them causes the evolution of **yellow chlorine tetroxide**— Cl_2O_4 , having a characteristic odor. When the dry salt is used this reaction takes place with **explosive violence**.

2. AgNO_3 produces no precipitate. This is an important distinction from HCl .

REACTIONS OF HYDROBROMIC ACID (HBr).

Use a solution of potassium bromide (KBr).

1. AgNO_3 produces a yellowish-white precipitate of **silver bromide**— AgBr , insoluble in HNO_3 , slowly soluble in NH_4OH .

2. $\text{Hg}_2(\text{NO}_3)_2$ precipitates yellowish-white **mercurous bromide**— Hg_2Br_2 .

3. H_2SO_4 , when concentrated, causes the evolution of red vapors of bromine; this occurs more readily in the presence of MnO_2 .

4. Chlorine water and starch paste cause a yellow color, due to formation of starch bromide; in dilute solutions it is necessary to agitate the mixture with ether or chloroform, which will separate, carrying the bromine in solution, with a red, or reddish-brown color.

REACTIONS OF HYDRIODIC ACID (HI).

Use a solution of potassium iodide (KI).

1. AgNO_3 produces a yellowish precipitate of **silver iodide**— AgI , insoluble in HNO_3 and almost insoluble in NH_4OH .

2. $\text{Hg}_2(\text{NO}_3)_2$ precipitates green **mercurous iodide**— Hg_2I_2 .

3. HgCl_2 causes a red precipitate of **mercuric iodide**— HgI_2 , soluble in excess of either reagent.

4. $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$ produces a yellow precipitate of **lead iodide**— PbI_2 , soluble in boiling water.

5. Chlorine water and starch paste form a blue color of starch iodide, which color disappears on heating and returns on cooling, also destroyed by excess of chlorine water.

6. A concentrated solution of one part CuSO_4 and three parts FeSO_4 produces a grayish precipitate of **cuprous iodide**— Cu_2I_2 . This reaction is useful in separating iodine from chlorine and bromine.

HYDROFLUORIC ACID (HF).

The evolution of intensely irritating fumes of this acid, on the addition of H_2SO_4 to calcium fluoride, which etch glass, is sufficiently characteristic.

REACTIONS OF HYDROCYANIC ACID (HCN).

Use a solution of potassium cyanide (KCN).

1. AgNO_3 produces a white precipitate of AgCN , soluble in KCN , sparingly soluble in NH_4OH , and insoluble in dilute HNO_3 .

2. $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ evaporated with KCN to dryness, on a water bath, will give on dissolving in water and adding Fe_2Cl_6 a deep blood red color, due to formation of sulphocyanate.

3. Fe_2Cl_6 and FeSO_4 , then NaOH until a precipitate is produced, heat and add HCl, will give a deep blue residue of **ferric ferrocyanide** (Prussian blue).

Hydroferrocyanic and Hydroferricyanic acids have been sufficiently characterized under iron.

WATER (H_2O), HYDRATES AND OXIDES.

Water is distinguished by having no odor or taste, not changing litmus, and evaporating without residue.

The soluble hydrates KOH, NaOH, LiOH, $\text{Ca}(\text{OH})_2$, $\text{Sr}(\text{OH})_2$, $\text{Ba}(\text{OH})_2$, yield solutions with water which change red litmus paper blue. The insoluble hydrates give off steam when heated in a dry tube.

The soluble oxides K_2O , Na_2O , Li_2O , CaO , SrO , BaO , are known by forming hydrates with water. The insoluble oxides are recognized by giving negative tests for acids when dissolved and tested in the usual way.

HYDROSULPHURIC ACID (H_2S).

Sufficient evidence of the presence of this acid is afforded by the characteristic odor. In the case of sulphides, first adding H_2SO_4 and warming if necessary. A trace may be detected by holding over the mouth of the tube a piece of filter paper moistened with lead acetate, which will become black (lead sulphide) in the presence of H_2S .

REACTIONS OF SULPHUROUS ACID (H_2SO_3).

1. In solution, uncombined, this acid is recognized by its odor of burning sulphur, by strong bleaching action, by decolorizing potassium permanganate solution, and by causing the evolution of hydrogen sulphide when added to a mixture of zinc and hydrochloric acid.

2. Sulphites are distinguished by the characteristic odor of SO_2 on the addition of a strong acid.

3. Salts of Ag, Hg or Pb, produce precipitates which blacken on heating, owing to formation of sulphides.

4. BaCl_2 with neutral solutions forms a white precipitate of **barium sulphite**— BaSO_3 , soluble in HCl .

REACTIONS OF SULPHURIC ACID (H_2SO_4).

Use dilute H_2SO_4 or an alkali sulphate.

1. BaCl_2 produces a white precipitate of **barium sulphate**— BaSO_4 , insoluble in boiling concentrated acids.

2. $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$ causes a precipitate of white **lead sulphate**, insoluble in dilute acids, but soluble in hot concentrated acids. Alcohol increases the delicacy of this reaction.

3. A sulphate fused on charcoal with Na_2CO_3 , the fused mass placed on a bright silver coin and moistened with a drop of dilute HCl will cause a black stain due to formation of silver sulphide. This reaction is especially adapted to the detection of *insoluble* sulphates.

REACTIONS OF THIOSULPHURIC (HYPOSULPHUROUS) ACID ($\text{H}_2\text{S}_2\text{O}_3$).

Use a solution of sodium thiosulphate ($\text{Na}_2\text{S}_2\text{O}_3$).

1. H_2SO_4 causes the evolution of **sulphurous oxide**— SO_2 , recognized by the odor. A deposit of sulphur takes place at the same time, which is an important distinction from sulphites.

2. AgNO_3 produces a white precipitate of **silver thio-sulphate**— $\text{Ag}_2\text{S}_2\text{O}_3$, soluble in excess. After a time (immediately on heating) the precipitate becomes dark and then black, silver sulphide and sulphuric acid being formed.

3. BaCl_2 produces a white precipitate soluble in excess of H_2O , and decomposed by HCl .

4. Added to a mixture of zinc and hydrochloric acid, hydrogen sulphide is evolved.

REACTIONS OF NITRIC ACID (HNO_3).

Use a solution of potassium nitrate (KNO_3).

1. H_2SO_4 on heating will cause the nitric acid to volatilize. If copper turnings be added with the sulphuric acid colorless

nitrogen dioxide— N_2O_2 will be given off, which from contact with air will form red **nitrogen tetroxide**— N_2O_4 , readily recognized by the color and odor. If alcohol be added to the mixture the characteristic odor of nitrous ether is developed.

2. FeSO_4 acidified with H_2SO_4 , added in a test tube, so as to form a layer on a solution of a nitrate, acidified with H_2SO_4 , will cause a dark layer to form at the line of contact.

3. Indigo solution strongly acidified with H_2SO_4 is decolorized by a nitrate.

4. Heated on charcoal, deflagration takes place, the charcoal burning at the expense of the oxygen of the nitrate.

HYPOPHOSPHOROUS ACID ($\text{H}_2\text{H}_2\text{PO}_2$).

1. By ignition the hypophosphites are resolved into spontaneously inflammable **hydrogen phosphide**— PH_3 , and phosphate.

2. AgNO_3 produces at first a white precipitate of **silver hypophosphite**— AgH_2PO_2 , which soon becomes black, owing to formation of metallic silver.

3. HgCl_2 in excess causes a white precipitate of mercurous chloride Hg_2Cl_2 , this takes place more rapidly on warming, and in the presence of HCl .

4. $(\text{NH}_4)_2\text{MoO}_4$ gives a blue precipitate; if this precipitate be green, it indicates a mixture of yellow caused by the presence of *phosphates*.

REACTIONS OF ORTHOPHOSPHORIC ACID (H_3PO_4).

Use a solution of sodium phosphate (Na_2HPO_4).

1. AgNO_3 causes a light yellow precipitate of **silver phosphate**— Ag_3PO_4 , soluble in HNO_3 and in NH_4OH .

2. Fe_2Cl_6 in presence of sodium acetate produces a yellowish-white, gelatinous precipitate of **ferric phosphate**— $\text{Fe}_2(\text{PO}_4)_2$. An excess of Fe_2Cl_6 must be avoided.

3. $(\text{NH}_4)_2\text{MoO}_4$ in neutral or acid solution causes a yellow precipitate to separate slowly, which is **ammonium phosphomolybdate**— $(\text{NH}_4)_3\text{PO}_4(\text{MoO}_3)_9 + 2\text{H}_2\text{O}$, insoluble in HNO_3 , soluble in NH_4OH .

4. Magnesia mixture (consisting of MgSO_4 , NH_4Cl , NH_4OH) causes a white precipitate of **ammonium magnesium phosphate**— $\text{MgNH}_4\text{PO}_4 + 6\text{H}_2\text{O}$. Agitation facilitates the formation of this precipitate.

5. BaCl_2 in neutral solution produces a white precipitate of **barium hydrogen phosphate**— BaHPO_4 .

6. Albumen (white of egg) does not cause a precipitate.

REACTIONS OF PYROPHOSPHORIC ACID ($\text{H}_4\text{P}_2\text{O}_7$).

Use a solution of sodium pyrophosphate ($\text{Na}_4\text{P}_2\text{O}_7$).

1. AgNO_3 precipitates white **silver pyrophosphate**— $\text{Ag}_4\text{P}_2\text{O}_7$, soluble in HNO_3 and NH_4OH .
2. MgSO_4 causes a white precipitate of **magnesium pyrophosphate**— $\text{Mg}_2\text{P}_2\text{O}_7$, soluble in excess of the reagent; from this solution NH_4OH does not reprecipitate it in the cold. This reaction may be used to distinguish ortho- from pyrophosphoric acid.
3. Neither $(\text{NH}_4)_2\text{MoO}_4$ nor albumen produces a precipitate.
4. BaCl_2 in neutral solution precipitates white **barium pyrophosphate**— $\text{Ba}_2\text{P}_2\text{O}_7$.

REACTIONS OF METAPHOSPHORIC ACID (HPO_3).

Use a solution of sodium metaphosphate (NaPO_3).

1. AgNO_3 produces a white precipitate of **silver metaphosphate**— AgPO_3 , soluble in HNO_3 and NH_4OH .
2. Albumen forms a white precipitate with the free acid, and with the salts on the addition of acetic acid. This is an important distinction from the ortho- and pyro-acids.
3. Neither $(\text{NH}_4)_2\text{MoO}_4$ nor magnesia mixture produces a precipitate; should one form with the latter reagent, it is readily soluble in NH_4Cl .
4. BaCl_2 forms a white precipitate of **barium metaphosphate**— $\text{Ba}(\text{PO}_3)_2$.
5. Solutions of the meta- and pyro-acids in water are converted into the ortho-acid by boiling.

REACTIONS OF BORIC ACID (H_3BO_3).

Use a solution of sodium borate ($\text{Na}_2\text{B}_4\text{O}_7$).

1. AgNO_3 produces a white precipitate of **silver borate**, soluble in HNO_3 .
2. BaCl_2 precipitates white **barium borate**, soluble in excess of water and in NH_4Cl .
3. H_2SO_4 or HCl causes the separation of the acid, H_3BO_3 , in crystalline form, from strong solutions.
4. Alcohol added to the acid, and ignited, burns with a

characteristic green flame. In the case of salts, the addition of alcohol is preceded by that of H_2SO_4 , in order to liberate the free acid. Salts of copper when present should be removed before this test is applied, as they likewise impart a green color to the alcohol flame.

REACTIONS OF CARBONIC ACID (H_2CO_3).

Use a solution of sodium carbonate (Na_2CO_3).

1. All free acids except HCN and H_2S decompose carbonates with effervescence. The escaping gas passed into a solution of $\text{Ba}(\text{OH})_2$ or $\text{Ca}(\text{OH})_2$ causes a white precipitate.
2. BaCl_2 in neutral solution precipitates white **barium carbonate**— BaCO_3 , soluble in acids.
3. CaCl_2 precipitates white **calcium carbonate**— CaCO_3 , soluble in acids with effervescence.

REACTIONS OF SILICIC ACID (H_4SiO_4).

1. Insoluble silicates are determined by fusing on platinum foil some of the fine powder with Na_2CO_3 , treating the fused mass with H_2O and HCl , evaporating to dryness and redissolving in H_2O , when the silica, SiO_2 , will remain as a fine white precipitate.
2. Before the blowpipe, with a bead of microcosmic salt, silica forms the so-called *silica skeleton*, which is very characteristic.
3. Soluble silicates give gelatinous precipitates of **silicic acid**— H_4SiO_4 , on the addition of H_2SO_4 or HCl ; on evaporating this to dryness with a little HCl , and redissolving in H_2O , *silica* SiO_2 remains.
4. NH_4Cl also precipitates H_4SiO_4 when added to a soluble silicate.

REACTIONS OF ACETIC ACID ($\text{HC}_2\text{H}_3\text{O}_2$).

1. In the free state acetic acid is readily recognized by its odor.
2. H_2SO_4 added to an acetate and the mixture warmed gives the characteristic odor.

3. H_2SO_4 and $\text{C}_2\text{H}_5\text{OH}$ in equal volumes added to an acetate form **acetic ether**, readily recognized by its odor.

4. Fe_2Cl_6 with a neutral acetate, forms a deep red color, due to **ferric acetate**— $\text{Fe}_2(\text{C}_2\text{H}_3\text{O}_2)_6$.

VALERIANIC ACID ($\text{HC}_5\text{H}_9\text{O}_2$).

The odor is sufficient evidence of the presence of this acid. This odor is developed by moisture and heat, and in the case of the salts by the addition of H_2SO_4 .

STEARIC ACID ($\text{HC}_{18}\text{H}_{35}\text{O}_2$).

Stearic acid is a white, fatty solid, melting at 70.5°C ., giving when combined with potassium a soft soap, and with sodium a hard soap; from both it separates as an oily liquid on the addition of HCl , becoming solid on cooling. The lead salt, lead stearate, $\text{Pb}(\text{C}_{18}\text{H}_{35}\text{O}_2)_2$ is insoluble in ether.

OLEIC ACID ($\text{HC}_{18}\text{H}_{33}\text{O}_2$).

Oleic acid is an oily liquid at ordinary temperatures, but becomes solid at 4°C . and remains so until the temperature rises to 14°C ., when it again becomes liquid. Lead Oleate— $\text{Pb}(\text{C}_{18}\text{H}_{33}\text{O}_2)_2$, prepared by neutralizing sodium oleate with acetic acid and adding lead acetate, is almost entirely soluble in ether. This is an important distinction from stearic acid.

LACTIC ACID ($\text{HC}_3\text{H}_5\text{O}_3$).

Lactic acid is a colorless, syrupy liquid, of a slight unpleasant odor and a very sour taste, soluble in water, alcohol and ether, but insoluble in chloroform. When heated with H_2SO_4 , CO is evolved. Lactates are all soluble in water, most of them sparingly; they are insoluble in ether.

Hg_2NO_3 boiled with strong solution of a lactate, deposits crimson **mercurous lactate**— $\text{Hg}_2(\text{C}_3\text{H}_5\text{O}_3)_2$.

REACTIONS OF OXALIC ACID ($\text{H}_2\text{C}_2\text{O}_4$).

Use a solution of ammonium oxalate ($(\text{NH}_4)_2\text{C}_2\text{O}_4$).

1. $\text{K}_2\text{Mn}_2\text{O}_8$ acidulated with H_2SO_4 is decolorized.
2. BaCl_2 produces a white precipitate of **barium oxalate**— BaC_2O_4 , sparingly soluble in acetic or oxalic acid, and freely soluble in HCl , HNO_3 and NH_4Cl .
3. AgNO_3 precipitates white **silver oxalate**— $\text{Ag}_2\text{C}_2\text{O}_4$, readily soluble in hot concentrated HNO_3 , sparingly so in dilute acid, soluble in NH_4OH .
4. CaCl_2 produces, even in highly diluted solutions, a white precipitate of **calcium oxalate**— CaC_2O_4 , soluble in HCl and HNO_3 , but insoluble in acetic acid.

SUCCINIC ACID ($\text{H}_2\text{C}_4\text{H}_4\text{O}_4$).*Use a solution of sodium succinate ($\text{Na}_2\text{C}_4\text{H}_4\text{O}_4$).*

1. BaCl_2 produces no precipitate, but on the addition of alcohol a white precipitate of **barium succinate**— $\text{BaC}_4\text{H}_4\text{O}_4$ falls, soluble in NH_4Cl .
2. Fe_2Cl_6 causes a brownish-red, bulky precipitate of **ferric succinate**— $\text{Fe}_2\text{C}_4\text{H}_4\text{O}_4\cdot 3$.
3. $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$ precipitates white amorphous **lead succinate**— $\text{PbC}_4\text{H}_4\text{O}_4$, soluble in HNO_3 .

MALIC ACID ($\text{H}_2\text{C}_4\text{H}_4\text{O}_5$).

1. CaCl_2 fails to give a precipitate with the acid or its salts, until the mixture is boiled, when **calcium malate**— $\text{CaC}_4\text{H}_4\text{O}_5\text{H}_2\text{O}$ separates. The addition of alcohol will also cause this precipitate.
2. $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$ precipitates white **lead malate**— $\text{PbC}_4\text{H}_4\text{O}_5\cdot 3\text{H}_2\text{O}$.

REACTIONS OF TARTARIC ACID ($\text{H}_2\text{C}_4\text{H}_4\text{O}_6$).*Use a solution of sodium tartrate ($\text{Na}_2\text{C}_4\text{H}_4\text{O}_6$).*

1. BaCl_2 precipitates white **barium tartrate**— $\text{BaC}_4\text{H}_4\text{O}_6$, soluble in ammonium salts, and in HCl .
2. CaCl_2 produces a white precipitate of **calcium tartrate**— $\text{CaC}_4\text{H}_4\text{O}_6\cdot 4\text{H}_2\text{O}$, ammonium salts prevent this precipitation. On adding KOH to this precipitate, it dissolves; boil this solution, calcium tartrate is again precipitated.
3. AgNO_3 causes a white precipitate of **silver tartrate**— $\text{Ag}_2\text{C}_4\text{H}_4\text{O}_6$; on boiling, a mirror of metallic silver forms on the test tube.
4. Tartaric acid and tartrates char on heating, and with H_2SO_4 the odor of burnt sugar is given off.

REACTIONS OF CITRIC ACID ($\text{H}_3\text{C}_6\text{H}_5\text{O}_7$).*Use a solution of sodium citrate ($\text{Na}_3\text{C}_6\text{H}_5\text{O}_7$).*

1. BaCl_2 produces a white precipitate of **barium citrate**— $\text{Ba}_3(\text{C}_6\text{H}_5\text{O}_7)_2$, soluble in excess of water, in ammonium salts, and in acids.
2. CaCl_2 precipitates white **calcium citrate**— $\text{Ca}_3(\text{C}_6\text{H}_5\text{O}_7)_2$, more insoluble in hot than in cold water, soluble in cold NH_4Cl , but insoluble in KOH .
3. AgNO_3 produces a white precipitate of **silver citrate**— $\text{Ag}_3\text{C}_6\text{H}_5\text{O}_7$; on boiling no metallic mirror is formed.
4. Citric acid and citrates char on heating, and with H_2SO_4 give off the odor of burnt sugar.

CARBOLIC ACID (C_6H_5OH).

1. HNO_3 with an aqueous solution of the acid forms a yellow color, due to **picric acid**— $C_6H_2(NO_2)_3OH$.
2. Fe_2Cl_6 produces a violet-blue color.
3. A piece of pine wood dipped in the acid, and then exposed to the fumes of HCl , becomes after a short time colored blue.
4. Bromine water causes a white flocculent precipitate.
5. Albumen is coagulated by the free acid.

BENZOIC ACID ($HC_7H_5O_2$).

1. Fe_2Cl_6 precipitates from neutral solutions flesh-colored **ferric benzoate**— $Fe_2(C_6H_5O_2)_6$, soluble in HCl , with separation of benzoic acid.
2. HCl causes the separation of benzoic acid from cold solutions of the benzoates.
3. $BaCl_2$ and $CaCl_2$ produce no precipitates with either the free acid or its salts.

SALICYLIC ACID ($HC_7H_5O_3$).

1. Fe_2Cl_6 produces a deep violet color, which is very characteristic.
2. Warmed with H_2SO_4 and methyl alcohol, the fragrant odor of methyl salicylate (oil of winter green) is developed.
3. HCl causes the separation of the free acid from cold solutions of the salicylates.

GALLIC ACID ($HC_7H_5O_5 \cdot H_2O$).

1. $FeSO_4$ produces no change.
2. Fe_2Cl_6 produces a bluish-black precipitate, which disappears on heating.
3. KOH , if not in excess, develops slowly a deep green color, which becomes red on the addition of acids. Alkaline carbonates cause the same green color, although more slowly.
4. No precipitate is produced with either gelatin or the alkaloids. With the former, however, a precipitation takes place when gum is present.

TANNIC ACID ($C_{14}H_{10}O_9$).

1. $FeSO_4$, when perfectly pure, causes no change; in the presence of oxygen, however, a dark color rapidly develops, which on standing, slowly becomes a precipitate.
2. Fe_2Cl_6 produces a bluish-black precipitate.
3. Normal solution of iodine mixed with a small quantity

of ammonia, previously diluted with ten times its volume of water, produces a brilliant red color. This reaction will take place with only traces of tannin.

4. Gelatin causes a white flocculent precipitate. This reaction is more delicate in the presence of small quantities of alum.

5. Alkaloids produce white precipitates, soluble in acetic acid and alcohol.

6. $K(SbO)C_4H_4O_8$ causes a white, gelatinous precipitate. Most metallic salts cause precipitates with tannin.

SECTION III.

DETECTION OF BASES AND ACIDS.

DIVISION I.—When the bases are the alkali metals, and the reaction is neutral.

RULE I.—*Evaporate a portion of the solution to dryness, and slowly heat to redness.*

If the mass chars, one or more of the following *organic acids* are indicated:—ACETATES, TARTRATES, CITRATES, GAL-LATES, TANNATES.

NOTE.—Oxalates do not char, although if the heating take place slowly a grayish coloration may be noticed, the residue in this case giving off CO_2 on the addition of H_2SO_4 .

RULE II.—*Add to a second portion of the concentrated solution, or the dry salt, strong H_2SO_4 , warm gently, and note any of the following effects:—*

Effervescence in the cold, no odor.....	Carbonates.
Effervescence on heating, no odor.....	{ Oxalates, confirm by CaCl_2 and $\text{HC}_2\text{H}_3\text{O}_2$.
Effervescence on heating, odor of H_2S	Sulphides.
Odor of SO_2	Sulphites.
Odor of SO_2 with precipitation of S.....	Thiosulphates.
Dark brown color and violet fumes.....	{ Iodides, confirm by starch and Cl water.
Dark red color and reddish fumes.....	{ Bromides, confirm by starch and Cl water.
Odor of HCN	Cyanides.
Odor of HCN with crystalline deposit.....	{ Ferro- or Ferri-cyanides, confirm by Fe_2Cl_6 and FeSO_4 .
Odor of acetic acid.....	Acetates.
Odor of chlorine.....	Hypochlorites or Chlorates.
Strongly acid suffocating fumes.....	Chlorides, confirm by AgNO_3 .
Strongly acid fumes becoming red when metallic Cu is added.....	{ Nitrates, confirm by FeSO_4 and H_2SO_4 , also by indigo solution.
Characteristic odors.....	{ Benzoates, Succinates, Valerianates, Carbolates, Hypophosphites.

RULE III.—*To a third portion add BaCl_2 .*

A white precipitate insoluble in HCl indicates SULPHATES.

RULE IV.—*To a fourth portion add CaCl_2 .*

A white precipitate soluble in excess of H_2O indicates SULPHATES; if insoluble in excess of H_2O and in acetic acid, OXALATES are indicated. A white precipitate soluble in KOH , reprecipitated on boiling, indicates TARTRATES; confirm by boiling with NH_4OH and AgNO_3 , forming a mirror of silver on the test tube.

A white precipitate soluble in NH_4Cl and reprecipitated on boiling indicates CITRATES. Citrates and Tartrates are also detected by heating some of the dry salt with H_2SO_4 , when the odor of burnt sugar is developed.

RULE V.—*To a fifth portion, acidified with HNO_3 , add AgNO_3 .*

A white, curdy precipitate, immediately and completely soluble in NH_4OH indicates CHLORIDES. If slowly soluble in NH_4OH BROMIDES may be present; confirm by chlorine water and starch.

A yellowish precipitate insoluble in NH_4OH IODIDES. A white precipitate soluble in strong, hot HNO_3 = CYANIDES. When bromides and iodides are present they may be separated, by adding to the original solution chlorine water and starch; continue the addition of chlorine water until the blue color of the starch iodide is discharged, then shake with chloroform; when a red color is imparted to the separated chloroform, Br is indicated.

RULE VI.—*Add to a sixth portion of the solution magnesia mixture.*

A white precipitate indicates PHOSPHATES and ARSENATES. In another portion separate the As by HCl and H_2S , and repeat the reaction for H_3PO_4 in the filtrate by neutralizing and adding magnesia mixture.

RULE VII.—*To a seventh portion add Fe_2Cl_6 .*

This reagent readily indicates TANNATES, GALLATES, FERRO- and FERRI-CYANIDES.

RULE VIII.—*To an eighth portion apply the special test with H_2SO_4 and FeSO_4 for NITRATES. And apply the special test for BORATES, by adding H_2SO_4 , alcohol, and igniting to obtain the green flame.*

DIVISION II.—When the solution is acid, or, if a solid, requires the use of HCl to dissolve it.

All the bases must be sought for in a portion of the solution. *If phosphates are present, determined by adding HNO_3 and $(\text{NH}_4)_2\text{MoO}_4$, the following chart must be followed in the separation of Group IV. If oxalates are present, heat the powder to redness, before beginning the analysis.*

DIRECTIONS FOR THE ANALYSIS OF GROUP IV WHEN PHOSPHATES ARE PRESENT.

In addition to Fe, Ce, Al, Cr, there may be present the phosphates of Ca, Sr, Ba, Mn, Mg. Dissolve the precipitate in HCl , add Na_2HPO_4 in excess, then excess of $\text{NH}_4\text{C}_2\text{H}_3\text{O}_2$; boil, filter.

Ppt. $\text{Fe}_2(\text{PO}_4)_3$ $\text{Al}_2(\text{PO}_4)_3$ ($\text{Ce}_2(\text{PO}_4)_3$). Wash with hot H_2O , then pour on the filter hot solution KOH .		Filt. Ba, Sr, Ca, Mn, Mg, Cr. (If green Cr is present.) Add K_2CrO_4 , warm, filter.	
Ppt. Fe, Ce. Wash, dissolve in HCl and test a portion with $\text{K}_4\text{Fe}(\text{CN})_6$ for Fe.	Filt. $\text{Al}_2(\text{PO}_4)_3$. Add $\text{HC}_2\text{H}_3\text{O}_2$ in excess, white ppt. = Al.	Ppt. Ba yellow.	Filt. Sr, Ca, Mn, Mg. Add very dilute H_2SO_4 , allow to stand, filter.
		Ppt. Sr. Confirm by flame test.	Filt. Ca, Mn, Mg. Add $(\text{NH}_4)_2\text{C}_2\text{O}_4$, filter.
		Ppt. Ca white.	Filt. Mg, Mn. Evaporate a portion to dryness and test with borax bead for Mn. To the remaining portion add Fe_2Cl_6 , filter out the $\text{Fe}_2(\text{PO}_4)_3$, to filtrate add NH_4Cl , NH_4OH and NH_4HS , to separate Fe and Mn, filter and test filtrate with Na_2HPO_4 , white ppt. = Mg.
To another por- tion add $\text{H}_2\text{C}_2\text{H}_3\text{O}_2$, then NH_4OH in excess and $(\text{NH}_4)_2\text{C}_2\text{O}_4$, white precipitate = Ce.			

A second portion of the substance is tested according to Rules I and II, DIVISION I.

A third portion is treated as follows:—

Boil with strong solution Na_2CO_3 ; filter.

Ppt. consists of the carbonates, hydrates, and oxides, of the bases present, and is disregarded.	Filt. contains the acids in combination with Na. Divide in two parts.	
	First portion. Add HCl until slightly acid, boil to remove CO_2 , neutralize with NaOH , and test according to Rules III, IV, VI, VII, and VIII. DIVISION I.	Second portion. Add HNO_3 until slightly acid, boil to remove CO_2 , neutralize with NaOH , and test according to Rule V, and if necessary, III, IV, VI, VII, DIVISION I.

DIVISION III.—When the substance is not entirely dissolved by H_2O , HCl , or a mixture of HCl and HNO_3 .

Add water and filter; the filtrate, if it contains solid matter, is set aside for examination by Division I. The residue after treatment with water is treated with HCl , or if necessary, with a mixture of HCl and HNO_3 , and, if it contain any solid matter, examined according to Division II. The insoluble residue consists of one or more of the following substances: Sulphates of Ba, Sr, Ca, Pb; Chlorides, Bromides and Iodides of Ag, Pb; certain Oxides which have been highly heated, as Fe_2O_3 , Al_2O_3 , Cr_2O_3 and SnO_2 ; Silica and Silicates; Carbon and Sulphur (the last two are readily detected in the preliminary examination by heat). This insoluble residue is mixed with about four times its weight of dry sodium carbonate and fused on a platinum foil; the mass is boiled with water, and the soluble portion examined according to Division I. The insoluble portion is treated with HCl and examined according to Division II. Any insoluble substance remaining is probably silica, which may be determined by a bead of salt of phosphorus before the blowpipe, showing the skeleton of silica. If the metals lead and silver are present in this insoluble residue, they are best determined by treating the residue insoluble in HCl , by HNO_3 and examining according to Division II. Many of these insoluble substances are detected in a preliminary examination by the use of the blowpipe, as explained in the tests given under the individual elements.

The following chart, taken largely from Dr. Muter's Analytical Chemistry, will be found useful to consult after determining the bases and before commencing the examination for acids.

Bases found.	If soluble in water test for the following acids.	If insoluble in water, but soluble in acids, test for the following acids.	If insoluble in acids, fuse with Na_2CO_3 and test for,
Ag,	$\text{HNO}_3, \text{HNO}_2, \text{H}_2\text{SO}_4, \text{HC}_2\text{H}_3\text{O}_2,$	Oxide, Sulphide, $\text{H}_2\text{CO}_3,$ $\text{H}_3\text{PO}_4, \text{HCN}, \text{H}_2\text{C}_2\text{O}_4,$ $\text{H}_2\text{C}_4\text{H}_4\text{O}_6, \text{H}_3\text{C}_6\text{H}_5\text{O}_7.$	Cl, I, Br.
Hg(ous),	$\text{HNO}_3, \text{HC}_2\text{H}_3\text{O}_2, \text{H}_2\text{SO}_4,$	Oxide, Sulphide, Cl, I, Oxysulphate.	Cl, I, Br.
Hg(ic),	Cl, $\text{HNO}_3, \text{H}_2\text{SO}_4, \text{HC}_2\text{H}_3\text{O}_2,$	Oxide, Sulphide, I, Oxysulphate.	Sulphide, Iodide.
Pb,	$\text{HC}_2\text{H}_3\text{O}_2, \text{HNO}_3,$	Oxide, Sulphide, $\text{H}_2\text{CO}_3,$ $\text{H}_3\text{PO}_4, \text{H}_2\text{C}_2\text{O}_4.$	$\text{H}_2\text{SO}_4, \text{Cl},$ I, $\text{H}_2\text{CrO}_4.$
Bi,	$\text{HNO}_3, \text{Cl}, \text{H}_2\text{SO}_4, \text{HC}_2\text{H}_3\text{O}_2,$	Oxynitrate, Oxychloride, Oxide, Sulphide, $\text{H}_2\text{CO}_3,$ $\text{H}_3\text{PO}_4.$	None.
Cu(ic),	Cl, $\text{HNO}_3, \text{H}_2\text{SO}_4, \text{HC}_2\text{H}_3\text{O}_2,$	Oxide, Sulphide, $\text{H}_2\text{CO}_3,$ $\text{H}_3\text{PO}_4, \text{Oxyacetate}.$	"
Cu(ous),	$\text{H}_2\text{SO}_4,$	Oxide, I.	"
Cd,	Cl, $\text{HNO}_3, \text{I}, \text{H}_2\text{SO}_4,$	Oxide, Sulphide, $\text{H}_2\text{CO}_3,$ $\text{H}_3\text{PO}_4.$	"
Sb,	Cl, $\text{H}_2\text{C}_4\text{H}_4\text{O}_6,$	Oxide, Sulphide, Oxychloride.	"
Sn(ic),	Cl,	Oxide.	"
Sn(ous),	Cl, $\text{H}_2\text{SO}_4,$	Oxide, Sulphide, $\text{H}_3\text{PO}_4,$ $\text{H}_2\text{CrO}_4.$	"
Au,	Cl,	Sulphide.	"
Pt,	Cl,	Sulphide.	"
Fer(ic),	Cl, $\text{HNO}_3, \text{H}_2\text{SO}_4, \text{HC}_2\text{H}_3\text{O}_2,$	Oxide, Sulphide, I, $\text{H}_3\text{PO}_4.$	"
Fer(ous),	Cl, $\text{H}_2\text{SO}_4, \text{I},$	Oxide, Sulphide, $\text{H}_2\text{CO}_3,$ $\text{H}_3\text{PO}_4.$	"
Al,	Like Iron,	Oxide, $\text{H}_3\text{PO}_4.$	"
Ce,	Cl,	Oxide, $\text{H}_2\text{C}_2\text{O}_4.$	Oxide.
Cr,	Cl, $\text{H}_2\text{SO}_4, \text{HC}_2\text{H}_3\text{O}_2, \text{HNO}_3,$	Oxide, $\text{H}_3\text{PO}_4.$	Oxide.
Mn,	Like Chromium,	Oxide, Sulphide, $\text{H}_2\text{CO}_3,$ $\text{H}_3\text{PO}_4.$	None.
Zn,	Like Chromium,	Like Manganese,	"
Co,	Cl, $\text{HNO}_3, \text{H}_2\text{SO}_4,$	Oxide, Sulphide, $\text{H}_2\text{CO}_3,$ $\text{H}_3\text{PO}_4.$	"
Ni,	Like Cobalt,	Like Cobalt.	"
Ba,	Cl, $\text{HNO}_3, \text{HC}_2\text{H}_3\text{O}_2,$	$\text{H}_2\text{CO}_3, \text{H}_3\text{PO}_4, \text{H}_2\text{C}_2\text{O}_4,$ $\text{H}_2\text{CrO}_4.$	Sulphate.
Sr,	Like Barium,	Like Barium.	"
Ca,	Like Barium,	Like Barium.	Sulphate.
Mg,	Oxide, Cl, $\text{H}_2\text{SO}_4,$	$\text{H}_2\text{CO}_3, \text{H}_3\text{PO}_4,$	None.
Li,	All Radicals,	$\text{H}_3\text{PO}_4,$	"
K,	All Radicals,	$\text{K}_2\text{PtCl}_6, \text{KHC}_4\text{H}_4\text{O}_6.$	"
Na,	All Radicals.	None.	"
$\text{NH}_4,$	All Radicals.	None.	"

SECTION IV.

SOME OF THE REACTIONS AND TESTS OF PURITY
OF THE MORE IMPORTANT ORGANIC
COMPOUNDS.CHLOROFORM (CHCl_3).

"A heavy, clear, colorless, diffusive liquid, of a characteristic, pleasant, ethereal odor, a burning, sweet taste, and a neutral reaction."

1. Agitate about 5 c.c. with twice its volume of distilled water, separate and test the aqueous portion with litmus paper; no change should be produced, showing the absence of acids. To a small portion of the water add AgNO_3 ; a white precipitate would indicate HCl , the result of decomposition. To another portion add solution of KI ; a reddish color would indicate *Chlorine*. Another portion warmed with solution KOH should not become colored, showing the absence of aldehyde.

2. 10 c.c. of chloroform mixed with half its volume of strong H_2SO_4 , and, after agitation, set aside, should not cause a color in either liquid for twenty-four hours.

3. H_2SO_4 and $\text{K}_2\text{Cr}_2\text{O}_7$ mixed with an equal volume of chloroform and warmed, will become green, indicating the presence of a small quantity of *alcohol*.

•
IODOFORM (CHI_3).

Iodoform occurs in small, lemon-yellow, lustrous crystals, having a saffron-like odor, and an unpleasant, slightly sweetish, iodine-like taste. Its solutions have a neutral reaction.

1. Digest a small quantity of iodoform with alcoholic solution of KOH , acidify with dilute HNO_3 , and add starch paste, when the blue color of starch iodide will be developed.

2. Agitate a small quantity of iodoform with distilled water and filter; the filtrate should not affect litmus paper. Add to a portion of the filtrate AgNO_3 ; a yellowish precipitate would

indicate iodides as an impurity. Evaporate another small portion to dryness; no residue should remain, indicating the absence of soluble impurities.

ALCOHOL (C_2H_5OH).

"A transparent, colorless, mobile and volatile liquid, of a characteristic, pungent and agreeable odor, and a burning taste. It should not change the color of blue litmus paper, previously moistened with water. It boils at $78^\circ C.$ and is readily inflammable, giving a blue flame without smoke."

1. Add to 5 c.c. of alcohol in a test tube an equal volume of H_2SO_4 to which has been added a small quantity of $K_2Cr_2O_7$, and warm; the odor of aldehyde will be developed, the liquid in the tube becoming green.

2. On heating 10 c.c. of alcohol with about one-fourth its volume of H_2SO_4 the odor of ether will be evolved.

3. Equal volumes of H_2SO_4 , acetic acid and alcohol, on warming, will give the odor of acetic ether. If much amyl alcohol be present the odor of amyl acetate may also be recognized.

4. Evaporate 10 c.c. of alcohol to about one-fifth its bulk, and add an equal volume of strong sulphuric acid; a reddish color will indicate amyl alcohol.

5. Mix in a test tube 5 c.c. of alcohol with an equal volume of solution KOH; an immediate darkening will indicate the presence of methyl alcohol, aldehyde or oak tannin.

CHLORAL HYDRATE (C_2Cl_3OH, H_2O).

(*Chloral*, U. S. P.)

Chloral hydrate occurs in colorless, transparent crystals, slowly vaporizing on exposure to the air, having an aromatic, penetrating and slightly acid odor, a bitterish caustic taste and a neutral reaction. It liquefies when mixed with carbolic acid or with camphor.

1. In a test tube heat to boiling a small quantity of chloral hydrate and water and add solution KOH; a vaporous milky mixture of chloroform results, readily recognized by its odor, while formate of potassium remains in solution.

2. To a hot aqueous solution of chloral hydrate in a test tube, add AgNO_3 , then NH_4OH and boil; a silver mirror is deposited on the tube.

3. To another portion of an aqueous solution add a few drops of HNO_3 and AgNO_3 ; no precipitate should be produced, indicating the absence of HCl .

4. Add NH_4HS to an aqueous solution; a reddish-brown coloration results, which on standing deposits a reddish-brown compound mixed with sulphur.

5. A crystal of chloral hydrate heated on platinum foil should volatilize without residue.

GLYCERIN ($\text{C}_3\text{H}_5(\text{OH})_3$).

"A clear, colorless liquid, of a syrupy consistence, oily to the touch, hygroscopic, without odor, very sweet and slightly warm to the taste, and neutral in reaction."

1. Make a borax bead in the loop of a platinum wire, moisten it with a small quantity of glycerin (previously rendered alkaline with a dilute solution of soda), and hold in the colorless flame of a bunsen burner; the flame is tinged green, owing to the liberation of boric oxide.

2. Warm 5 c.c. of glycerin with an equal volume of H_2SO_4 ; no coloration should result, showing the absence of cane sugar.

3. Heat a few drops on platinum foil; no residue should be left.

4. A portion heated nearly to the boiling point with an equal volume of Fehling's solution, should not deposit a red precipitate of cuprous oxide Cu_2O . The same result should be obtained if the glycerin be previously boiled with a small quantity of HCl , showing the absence of sugars, starch and dextrin.

GLUCOSE ($\text{C}_6\text{H}_{12}\text{O}_6$).

(*Dextrose, Grape Sugar.*)

1. To a dilute solution of glucose, add an equal volume of Fehling's solution and boil; a red precipitate of cuprous oxide, Cu_2O , is deposited.

2. A strong solution is mixed with H_2SO_4 in the cold; no change should occur; if, however, organic impurities are present, a dark coloration will result.

3. To a strong solution of glucose add a solution of KOH and warm; the solution becomes yellow and finally brown.

SACCHAROSE ($\text{C}_{12}\text{H}_{22}\text{O}_{11}$).

(*Cane Sugar.*)

1. Boil a moderately dilute solution of sugar with a few drops of HCl , neutralize and add Fehling's solution; red cuprous oxide, Cu_2O , will be precipitated. If the sugar be pure this reaction will not take place unless it be previously boiled with the acid.

2. To a cold saturated solution add an equal volume of concentrated H_2SO_4 ; there is an immediate blackening and swelling until the tube is filled with a dry coke.

AMYLOSE ($\text{C}_6\text{H}_{10}\text{O}_5$) $_x$.

(*Starch.*)

Starch usually occurs as a fine, white powder, insoluble in cold water, soluble in boiling water, forming a thick paste when cold.

1. To a cold dilute solution add potassium iodide and a few drops of chlorine water; the deep blue color of starch iodide results. This is decolorized by heating and reappears on cooling. It is bleached by excess of chlorine water.

2. Heat a small quantity of starch on a platinum foil; it should char and finally disappear without residue.

3. Triturate a small quantity of starch with cold water, filter, and test the filtrate with litmus; no change should take place.

MORPHINE ($\text{C}_{17}\text{H}_{19}\text{NO}_3, \text{H}_2\text{O}$).

1. To some crystals of morphine sulphate on a crucible lid, or other white porcelain surface, add a small quantity of neutral solution of ferric chloride; a blue color is produced, which rapidly fades. The presence of free sulphuric acid must be avoided.

2. Similarly, to another portion add a drop of strong HNO_3 ; a red color at first forms, which rapidly becomes yellow.

3. To a solution of the sulphate add carefully a dilute solution of KOH ; a white precipitate forms, which is readily soluble in excess. NH_4OH and Na_2CO_3 produce the same precipitate, *insoluble* in excess.

4. A few crystals on a white porcelain surface should entirely dissolve in strong H_2SO_4 without color, becoming reddish on standing, and on the addition of a crystal of $\text{K}_2\text{Cr}_2\text{O}_7$ a greenish color is produced. A purple or violet color would indicate the presence of strychnine or brucine.

STRYCHNINE ($\text{C}_{21}\text{H}_{22}\text{N}_2\text{O}_2$).

1. A small quantity of the alkaloid dissolved in a drop or two of H_2SO_4 , and a small crystal of $\text{K}_2\text{Cr}_2\text{O}_7$ added will cause a deep blue color, rapidly changing to violet, then cherry-red, and finally fading.

2. Strong HNO_3 added to strychnine should not cause more than a very faint red color, showing the absence of brucine.

3. KOH added to a solution of the sulphate causes a white precipitate, *insoluble* in excess.

QUININE ($\text{C}_{20}\text{H}_{24}\text{N}_2\text{O}_2, 3\text{H}_2\text{O}$).

1. To an aqueous solution of the sulphate add fresh chlorine water and then NH_4OH in slight excess, when a green color is formed, due to *Thalleioquin*.

2. To a very dilute, slightly acid solution, add chlorine water, then a small quantity of $\text{K}_4\text{Fe}(\text{CN})_6$, and finally a few drops of NH_4OH ; a red color is produced, which rapidly fades.

3. Add KOH to an aqueous solution of the sulphate; a white precipitate forms, *insoluble* in excess. Under the same circumstances NH_4OH produces a white precipitate, *soluble* in excess.

CINCHONIDINE ($\text{C}_{20}\text{H}_{24}\text{N}_2\text{O}$).

1. To an aqueous solution of the sulphate add NH_4OH ; a white precipitate forms, almost insoluble in excess.

2. $\text{KNaC}_4\text{H}_4\text{O}_6$ added to a neutral solution produces a white precipitate.

3. To a cold saturated solution of the sulphate add $\text{KNaC}_4\text{H}_4\text{O}_6$ in slight excess, allow to stand a short time at about 15°C ., filter and to the filtrate add a drop of NH_4OH ; not more than a slight turbidity should result, showing the absence of more than small quantities of the other cinchona alkaloids.

CINCHONINE ($\text{C}_{20}\text{H}_{24}\text{N}_2\text{O}$).

1. To a solution of the sulphate add NH_4OH ; a white precipitate results, *insoluble* in excess.

2. To a saturated solution add chlorine water and then NH_4OH in slight excess; a white precipitate is formed, free from green color.

ATROPINE ($\text{C}_{17}\text{H}_{23}\text{NO}_3$).

1. Add to a small quantity of the alkaloid a drop of H_2SO_4 , no change occurs; add to the mixture HNO_3 , no color is produced, showing the absence of and difference from morphine; similarly when a crystal of $\text{K}_2\text{Cr}_2\text{O}_7$ is added no change occurs, indicating the absence of and difference from strychnine.

2. To a small quantity of atropine in a test tube add H_2SO_4 and heat until it turns brown; dilute with water and boil, when the characteristic odor of orange flowers will be developed.

3. Boil with $\text{K}_2\text{Cr}_2\text{O}_7$ and dilute H_2SO_4 , and add KOH in excess, when a herring-like odor will be given off.

CAFFEINE ($\text{C}_8\text{H}_{10}\text{N}_4\text{O}_2, \text{H}_2\text{O}$).

1. To an aqueous solution of the alkaloid add a solution of potassio-mercuric iodide; no precipitation occurs, indicating the absence of and distinction from most other alkaloids.

2. Treat a small quantity with chlorine water and evaporate to dryness, when a yellow mass will remain, which on moistening with NH_4OH will become purple.

VERATRINE.

1. To a small quantity on a crucible lid, add a drop of HNO_3 ; it dissolves with a yellow color, which soon passes to a reddish-yellow, then to an intense scarlet and finally to a violet-red.

2. Similarly, to another portion add H_2SO_4 ; very little change takes place, but on warming a resinous mass is formed, which dissolves with a deep red color.

3. Repeat the above reaction, using HCl , when a blood-red color results.

SALICIN ($\text{C}_{13}\text{H}_{18}\text{O}_7$).

1. Add cold, concentrated H_2SO_4 when a red color is produced, which disappears on adding water, a dark red powder insoluble in water and in alcohol being deposited.

2. The aqueous solution of salicin should not be precipitated by tannic or picric acids, nor potassio-mercuric iodide, indicating the absence of and difference from most alkaloids.

SANTONIN ($\text{C}_{15}\text{H}_{18}\text{O}_3$).

1. To an alcoholic solution of KOH add santonin; it will dissolve with a red color, gradually becoming colorless.

2. To another portion of santonin add cold, concentrated H_2SO_4 ; there is gradually formed a yellow color, becoming red and finally brown.

3. Santonin should give no precipitates with the ordinary reagents for the alkaloids.

NOTE.—*All the preceding organic compounds may be tested for mineral impurities by heating on a platinum foil; no residue should be left.*

PART THIRD.

QUANTITATIVE ANALYSIS.

PART III.—QUANTITATIVE CHEMICAL ANALYSIS.

SECTION I.

GRAVIMETRIC ESTIMATION.

PRELIMINARY DIRECTIONS.

The following course supposes the student to be familiar with the operations in *qualitative analysis*, as well as with the process of weighing, or in a position to be instructed by some one familiar with the use of analytical balances and weights. The following rules, however, should be kept constantly before those using a fine balance:—

1. *Never put any chemical substance directly on the pan of a balance, but always in a clean, dry watch crystal.*
2. *Never put on or take off a weight, or anything else, from a balance when it is resting on the knife edges.*
3. *All volatile acids and other corrosive substances should be weighed in stoppered weighing tubes.*
4. *Do not keep the balance open longer than is absolutely necessary.*

PRECIPITATION.

This should be conducted in beaker glasses. Avoid a large excess of the reagent, except in a few cases where it is directed. The filtrate should always be tested with a few drops more of the reagent, to determine that the precipitation has been complete. Distilled water is used in all quantitative determinations.

FILTRATION.

A filter paper having a very small ash should be used; the Swedish is probably the best. Plain filters are always used, made by folding a circular piece of the paper twice, and opening so as to form a cone, which will fit exactly into a funnel, and allow all the liquid to pass through the precipitate and

escape at the lower point into the neck of the funnel. The precipitate should be allowed to settle in the beaker after precipitation; the clear supernatant liquid is then poured on the filter, using a glass rod against the edge of the beaker, to conduct it into the funnel without loss. The precipitate is then washed into the filter with the aid of the wash bottle, and the washing continued, until by the appropriate test it is found to be complete. The washing, in many cases, is to be done with hot water.

DRYING.

The filter and its contents are usually dried in the funnel when intended for ignition, but if to be weighed without burning, it is transferred to two watch crystals clamped together, and dried in an air bath, at from 120° to 130° C., until it ceases to lose weight.

IGNITION.

This is usually done in a platinum or porcelain crucible, the precipitate being separated as much as possible from the filter, and the latter burned on the lid, then placed in the crucible with the precipitate and the whole brought to a low or bright red heat, as the case requires. It is then cooled in a desiccator, until nearly the temperature of the balance room, when it is ready for weighing.

Examples for Practice by Gravimetric Estimation.

BARIUM CHLORIDE ($\text{BaCl}_2\text{H}_2\text{O}$).

Estimation of the Barium.

Weigh about .500 gram of the pure salt, dissolve in 100 c.c. of water in a beaker of about 200 c.c. capacity. Add dilute sulphuric acid, drop by drop, as long as any precipitate is produced, and then boil the mixture. Allow the precipitate to settle, pour the clear supernatant liquid into a filter, boil with a fresh quantity of water, and after pouring in the clear liquid, transfer the precipitate to the filter. Remove the small part of the precipitate which adheres to the sides of the beaker with a glass rod having a short section of rubber tubing slipped

over the end. Wash the precipitate with hot water, until the washings cease to cause a turbidity with solution of barium chloride, then dry, ignite and weigh as barium sulphate, BaSO_4 .

The following calculation will serve to illustrate the general method:—

$$\begin{array}{rcl}
 \text{Ba} & = & 136.8 = 56.16 \text{ per cent.} \\
 \text{Cl}_2 & = & 70.8 = 29.06 \text{ " } \\
 2\text{H}_2\text{O} & = & 36. = 14.78 \text{ " } \\
 \hline
 & & 243.6 \quad 100.00
 \end{array}$$

Supposing we take .365 gram of barium chloride and find .349 gram of barium sulphate, we have as—

Molecular wt. of BaSO_4 .		Atomic wt. of Ba.		Wt. of BaSO_4 found.		Wt. of Ba found.
232.8	:	136.8	:	.349	:	.205 +
Wt. of $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ taken.		Wt. of Ba found.				Per cent. of Ba found.
As .365	:	.205 +	:	100	:	56.16.

ESTIMATION OF THE CHLORINE.

To accomplish this weigh another portion of the salt, dissolve in about 100 c.c. of water, acidify with HNO_3 , add AgNO_3 in slight excess and heat to the boiling point. Stir the mixture while hot, until the precipitate coagulates, allow to settle, decant the clear liquid to a filter, then rapidly transfer the precipitate, and wash it well with hot water, dry and ignite, burning the filter separately. If an appreciable amount of the precipitate adheres to the filter it is best to moisten the residue after burning with a drop or two of nitrohydrochloric acid, to reconvert any metallic silver which may have formed into chloride. The whole operation should be conducted as rapidly as possible, as silver chloride is acted on by the light. The calculation is performed precisely like that for barium, substituting Cl_2 for Ba, and AgCl for BaSO_4 .

COPPER SULPHATE ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$).

Estimation of the Copper.

Weigh from .500 to 1. gram, dissolve in a small quantity of water in a porcelain dish; add potassium hydrate in slight excess and boil, to convert the cupric hydrate first formed into

cupric oxide. Wash two or three times by decantation, then transfer to the filter and again wash with hot water until the filtrate ceases to change red litmus paper. The precipitate is separated from the filter, and the latter after burning is moistened with a drop of nitric acid and again heated, to reconvert any metallic copper which may have been formed; this residue is then transferred to the crucible with the precipitate and the whole ignited and weighed as cupric oxide CuO .

ESTIMATION OF SULPHURIC ACID.

Weigh another portion of the copper sulphate, dissolve and precipitate the sulphuric acid with barium chloride. Treat the precipitate exactly as in the estimation of barium.

POTASSIUM NITRATE (KNO_3).

Estimation of the Potassium.

To a weighed portion of the salt dissolved in a small quantity of water, add hydrochloric acid, and evaporate to dryness, dissolve in a small quantity of water, add platinic chloride in excess and evaporate nearly to dryness on a water bath, keeping the water in the bath just below the boiling point. Add 80 per cent. alcohol to the residue, allow to stand some time, then transfer to a small weighed filter, and wash repeatedly with small quantities of the same strength alcohol. Dry the precipitate first in the air then in an air bath at 130°C ., and weigh between two watch crystals. From the potassium platinic chloride (K_2PtCl_6) the potassium is readily calculated.

ESTIMATION OF THE NITRIC ACID.

Fuse in a platinum crucible, at a low temperature, some potassium nitrate, and pour out on a warm porcelain slab. Ignite two or three grams of silica in the crucible and mix with it about one-fourth its weight of the fused and powdered nitre. Bring the mixture to a low red heat and keep at that temperature until it ceases to lose weight. The loss indicates the amount of N_2O_5 from which the HNO_3 can be calculated. Sulphates and chlorides are not decomposed at this temperature.

CALCIUM CARBONATE (CaCO_3).*Estimation of the Calcium.*

Dissolve a weighed quantity of the carbonate in dilute hydrochloric acid, avoiding excess of acid, add ammonium hydrate until slightly alkaline, then ammonium oxalate, and set aside for twelve hours. Decant the clear supernatant liquid to a filter, wash once or twice by decantation, transfer the precipitate to the filter and wash with small portions of water, allowing each portion to completely run through before adding more. Dry and ignite the precipitate, using a strong Bunsen burner with a chimney, so as to bring the crucible to a white heat, until it ceases to lose weight; if this cannot be effected with a Bunsen burner, use a blast lamp. The residue is weighed as calcium oxide, CaO .

ESTIMATION OF THE CARBONIC ACID.

This in some cases is done by ignition, but the method adapted to all cases is to treat the carbonate with an acid, as HCl or H_2SO_4 , and estimate the loss of CO_2 . This is best accomplished by using a *carbonic acid apparatus*, that known as **Geissler's** is probably the best.

An apparatus may be constructed, however, by taking a small, wide mouth flask, and adapting to it a small tube containing calcium chloride. A weighed quantity of the material is then placed in the flask with some water, a test tube containing sulphuric acid is put in so as to retain the acid until the cork with the drying tube is fixed in place, and the whole apparatus weighed.

The flask is then turned over so as to allow some of the acid to flow out, and this is repeated, allowing only a small quantity to run out at a time, until the carbonate has been completely decomposed. The flask is then heated, to drive out all the CO_2 , and after cooling the apparatus is weighed. The loss indicates the amount of CO_2 in the material used.

SECTION II.

VOLUMETRIC ESTIMATION.

Volumetric analysis is the process of determining the amount of a substance by the use of a suitable reagent in solution of a known strength. The requisite apparatus consists of a *liter flask* and a *1000 cubic centimeter cylinder*, in which to prepare the solutions. A *100 c.c. burette* (Geissler's is the most desirable) with a suitable holder. A few *pipettes*, assorted sizes, some of which are graduated to $\frac{1}{10}$ c.c. The simplest process is that by *neutralization*, where an alkali is determined by neutralizing it with standard solution of oxalic acid. In such a case it is necessary to have a means of knowing when the alkali is exactly neutralized. Such a substance is known as an *indicator*. Solution of litmus may be used, but a solution of phenolphthalein gives most satisfactory results; it is colorless in neutral or acid and bright red in alkaline solution. It is made by dissolving 1 part phenolphthalein in 25 parts alcohol and adding sufficient water to make 100 parts.

A *normal solution* contains in every liter the molecular weight of the compound in grams. This is sometimes made *semi-normal*, when the compound, an acid for instance, is dibasic.

A *decinormal solution* is one-tenth the strength of the normal one. While in some of the following examples the normal solution is used, that of decinormal strength is recommended for the use of students.

VOLUMETRIC SOLUTION OF OXALIC ACID,
($\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O} = 126.$)

The seminormal solution is used, containing 63 grams in 1 liter.

Take of oxalic acid carefully purified by crystallization 63 grams, transfer it to a liter flask, add about 800 c.c. of water, agitate until dissolved and bring the measure, with water, to 1000 c.c.

ESTIMATION OF POTASSIUM HYDRATE.

Dissolve a convenient quantity of potassium hydrate, for instance .500 gram in a beaker containing 100 c.c. of water; add three or four drops of the phenolphthalein solution; set the beaker on a piece of white paper and bring over it a burette containing some of the oxalic acid solution. Allow the acid solution to run in slowly, constantly stirring with a glass rod, until the red color just ceases to be visible. The proof of this is that one drop of a similar alkali solution will restore the red color. If the potassium hydrate were pure, it would be found that 8.94 c.c. were used, but if we suppose that 8.50 c.c. were necessary, we have every c.c. of the oxalic solution = .056 gram of KOH, therefore $8.50 \times .056 = .476$ gram—the amount of KOH in the .500 gram = 95.20 per cent.

One c.c. containing 0.063 gram of oxalic acid is the equivalent of:—

	GRAM.
Ammonia, NH_3	0.0170
Ammonium Carbonate, $\text{NH}_4\text{HCO}_3\text{NH}_4\text{NH}_2\text{CO}_2$	0.0523
Potassium Acetate, $\text{KC}_2\text{H}_3\text{O}_2$ (after ignition).....	0.0980
Potassium Bicarbonate, KHCO_3	0.1000
Potassium Carbonate, (dry) K_2CO_3	0.0690
Potassium Citrate, $\text{K}_3\text{C}_6\text{H}_5\text{O}_7\text{H}_2\text{O}$ (after ignition).....	0.1080
Potassium Hydrate, KOH.....	0.0500
Potassium Permanganate, $\text{K}_2\text{Mn}_2\text{O}_8$	0.0314
Potassium Sodium Tartrate, $\text{KNaC}_4\text{H}_4\text{O}_6\text{H}_2\text{O}$ (after ignition).....	0.1410
Potassium Tartrate, $(\text{K}_2\text{C}_4\text{H}_4\text{O}_6)_2\text{H}_2\text{O}$ (after ignition).....	0.1175
Lead Acetate, $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2\text{H}_2\text{O}$	0.1802
Lead Subacetate, $\text{Pb}_2\text{O}(\text{C}_2\text{H}_3\text{O}_2)_2$	0.1367
Sodium Bicarbonate, NaHCO_3	0.0840
Sodium Borate, $\text{Na}_2\text{B}_4\text{O}_7\text{H}_2\text{O}$	0.1910
Sodium Carbonate, crystallized, $\text{Na}_2\text{CO}_3\text{H}_2\text{O}$	0.1430
Sodium Carbonate, anhydrous, Na_2CO_3	0.0530
Sodium Hydrate, NaOH.....	0.0400

When carbonates are estimated it is necessary to boil, towards the end of the reaction, after each addition of the acid, in order to drive off the CO_2 , which affects the indicator.

VOLUMETRIC SOLUTION OF SODIUM HYDRATE (NaOH = 40).

The normal solution is used, containing 40 grams in 1 liter.

As sodium hydrate is never, or very rarely absolutely pure, it is necessary to standardize this solution. For this take something more than the theoretical amount (about 50 grams) and dissolve in a liter of water. Place 100 c.c. of the standard oxalic acid solution in a beaker, and, having added the indicator, bring it under a burette containing some of the soda solution, and note the number of c.c. necessary to exactly neutralize the acid solution. Take of the alkaline solution ten times the number of c.c. necessary to neutralize the 100 c.c. of the acid solution, and add sufficient water to bring the measure to 1000 c.c. For instance, if it required 95 c.c. of the alkaline solution to neutralize the 100 c.c. of the oxalic acid solution, then $95 \text{ c.c.} \times 10 = 950 \text{ c.c.}$ which amount diluted to 1000 c.c. would make it exactly equal in strength to the oxalic acid solution.

One c.c. containing 0.040 of sodium hydrate NaOH, is the equivalent of:—

	GRAM.
Acetic Acid, absolute, $\text{HC}_2\text{H}_3\text{O}_2$	0.0600
Citric Acid, crystallized, $\text{H}_3\text{C}_6\text{H}_5\text{O}_7\text{H}_2\text{O}$	0.0700
Hydrobromic Acid, absolute, HBr.....	0.0808
Hydrochloric Acid, absolute, HCl	0.0364
Hydriodic Acid, absolute, HI.....	0.1276
Lactic Acid, absolute, $\text{HC}_3\text{H}_5\text{O}_3$	0.0900
Nitric Acid, absolute, HNO_3	0.0630
Oxalic Acid, crystallized, $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$..	0.0630
Sulphuric Acid, absolute, H_2SO_4	0.0490
Tartaric Acid, crystallized, $\text{H}_2\text{C}_4\text{H}_4\text{O}_6$	0.0750

VOLUMETRIC SOLUTION OF POTASSIUM BICHROMATE ($\text{K}_2\text{Cr}_2\text{O}_7 = 294.8$).

A viginti-normal solution is used, containing 14.74 grams in 1 liter.

Place the necessary amount of the salt (14.74 grams) in a liter flask, add about 800 c.c. of water, agitate until dissolved, and bring the measure with water to 1000 c.c. This solution is used in the estimation of iron in the ferrous condition. The end of the reaction is determined by taking out a drop

of the iron solution, and testing on a white porcelain surface with a drop of potassium ferricyanide solution; when this ceases to give a blue color the reaction is at an end.

One c.c. containing 0.01474 gram of potassium bichromate, $K_2Cr_2O_7$, is the equivalent of:—

	GRAM.
Iron in ferrous condition, Fe.....	0.01677
Ferrous Carbonate, $FeCO_3$	0.03477
Ferrous Sulphate, $FeSO_4 \cdot 7H_2O$	0.08337
Ferrous Sulphate, (dry) $FeSO_4 \cdot H_2O$	0.05097

VOLUMETRIC SOLUTION OF IODINE ($I = 126.6$).

A decinormal solution is used, containing 12.66 grams in 1 liter.

Weigh the necessary amount of iodine (12.66 grams) in a stoppered tube, to prevent loss, as well as the corrosive action of the fumes on the balance. Also weigh 18 grams of potassium iodide and place in a liter flask with the iodine. Add about 200 c.c. of water. The iodine dissolves more readily in this strength of potassium iodide solution, besides it admits of more thorough agitation. When the solution is complete add water until the liquid measures 1000 c.c. Starch solution is used as an indicator in the determinations with the iodine solution.

One c.c. containing 0.01266 iodine is the equivalent of:—

	GRAM.
Arsenious Oxide, As_2O_3	0.00494
Potassium Sulphite, crystallized, $K_2SO_3 \cdot 2H_2O$	0.0097
Sodium Bisulphite, $NaHSO_3$	0.0052
Sodium Hyposulphite, crystallized, $Na_2S_2O_3 \cdot 5H_2O$	0.0248
Sodium Sulphite, crystallized, $Na_2SO_3 \cdot 7H_2O$	0.0126
Sulphurous Oxide, SO_2	0.0032

VOLUMETRIC SOLUTION OF SODIUM HYPOSULPHITE ($Na_2S_2O_3 \cdot 5H_2O = 248$).

A decinormal solution is used, containing 24.8 grams in 1 liter.

Sodium hyposulphite cannot be prepared sufficiently pure to be relied on, consequently this solution must be standardized, therefore more than the theoretical amount is taken. The U. S. Pharmacopœia recommends 32 grams. Dissolve this amount in 1000 c.c. of water; place 100 c.c. of the standard solution of iodine in a beaker and run in the hyposulphite

solution until the color of iodine nearly disappears, then add a small quantity of starch solution and continue until the blue color is discharged. Multiply the number of cubic centimeters of the hyposulphite solution used by 10, and to that amount add sufficient water to bring the measure to 1000 c.c. The substances estimated by this solution either contain free iodine, or develop it on the addition of potassium iodide, so that starch solution may be used as an indicator.

One c.c. containing 0.0248 gram of sodium hyposulphite is the equivalent of:—

	GRAM.
Bromine, Br.....	0.00798
Chlorine, Cl.....	0.00354
Iodine, I	0.01266

VOLUMETRIC SOLUTION OF SILVER NITRATE ($\text{AgNO}_3 = 169.7$).

A decinormal solution is used, containing 16.97 grams in 1 liter.

As silver nitrate can be obtained or prepared perfectly pure, the necessary amount (16.97 grams) is dissolved in sufficient distilled water to make 1000 c.c.

In testing one of the following compounds, ammonium chloride for instance, a weighed amount is taken, dissolved in water, and a few drops of potassium bichromate solution added. The silver nitrate solution is then run in until a red precipitate remains permanently. The silver combines with the chlorine until the latter is all used, when it forms with the chromic acid red silver chromate, so that its formation indicates the end of the reaction.

One c.c. containing 0.01697 gram of silver nitrate is the equivalent of:—

		GRAM.
Ammonium Bromide,	NH_4Br	0.00978
Ammonium Chloride,	NH_4Cl	0.00534
Ferrous Bromide,	FeBr_2	0.01077
Ferrous Iodide,	FeI_2	0.01545
Hydrocyanic Acid, absolute,	HCN	0.00270
Hydriodic Acid,	HI	0.01276
Potassium Bromide,	KBr	0.01188
Potassium Chloride,	KCl	0.00744
Potassium Cyanide,	KCN	0.01300
Sodium Bromide,	NaBr	0.01028
Sodium Chloride,	NaCl	0.00584

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